Base-mediated formation of amides from aliphatic amines and ynones as acylation agents via C–C bond cleavage at room temperature

Guolin Cheng,*[a] Weiwei Lv,[a] Changsheng Kuai,[a] Si Wen[a] and Shangyun Xiao[a]

[a]College of Materials Science & Engineering, Huaqiao University, Xiamen 361021, China

Table of Contents

1. General Information S2

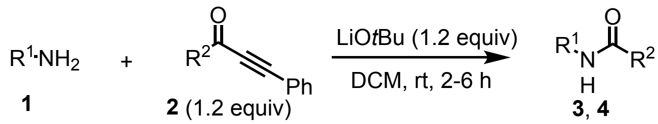
2. General Procedure for Synthesis of 3a-l, 4a-u. S3

3. ^{1}H and ^{13}C NMR Spectra S10

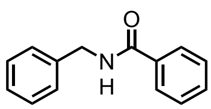
1. General Information

All reagents were used directly without further purification. Silica gel was purchased from Qing Dao Hai Yang Chemical Industry Co. ^{1}H and ^{13}C NMR spectra were measured on a 400 MHz Bruker spectrometer (^{1}H 400 MHz, ^{13}C100 MHz), using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. HRMS-ESI spectra were obtained on Agilent 6450 spectrometer. The products listed below were determined by ^{1}H, ^{13}C NMR. PE is petroleum ether (60–90 ^{o}C).

2. General Procedure for Synthesis of 3a-l, 4a-u.

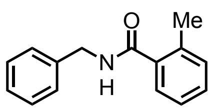


LiO*t*Bu (20 mg, 0.24 mmol) was added to a solution of amines **1** (0.2 mmol) in dry dichloromethane (1.0 mL). The solution was stirred for 5 min at room temperature, then ynones **2** (0.24 mmol) was added. The solution was stirred for another 2-6 h at room temperature. After the reaction finished, the organic layer was washed with saturated aqueous sodium chloride (2.0 mL), dried over sodium sulfate, filtered and concentrated. The residue was purified by column chromatography (ethyl acetate/PE = 1/4) to yield the desired amides. Note: in the case of **4q**, ynones **2a** (0.48 mmol) was used.



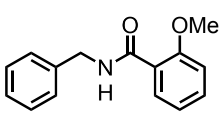
***N*-benzylbenzamide (3a)[1]**

Substrate **3a** was obtained as white powder (34.6 mg, 82%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.6 Hz, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.24 (m, 5H), 6.58 (s, 1H), 4.62 (d, *J* = 5.7 Hz, 2H);^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 138.2, 134.3, 131.5, 128.7, 128.5, 127.8, 127.5, 126.9, 44.1.



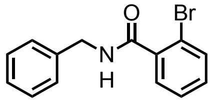
***N*-benzyl-2-methylbenzamide (3b)[1]**

Substrate **3b** was obtained as white powder (22.5 mg, 50%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.25 (m, 7H), 7.22 – 7.15 (m, 2H), 6.08 (s, 1H), 4.61 (d, *J* = 5.7 Hz, 2H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 138.2, 136.2, 136.2, 131.0, 129.9, 128.8, 127.8, 127.6, 126.6, 125.7, 43.9, 19.8.



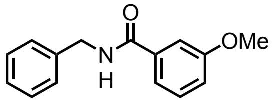
***N*-benzyl-2-methoxybenzamide (3c)[2]**

Substrate **3c** was obtained as white powder (34.3 mg, 71%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 – 8.22 (m, 1H), 8.19 (s, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.41 – 7.23 (m, 5H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 8.3 Hz, 1H), 4.69 (d, *J* = 5.7 Hz, 2H), 3.91 (s, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 157.5, 138.8, 132.8, 132.4, 128.6, 127.5, 127.2, 121.4, 121.2, 111.3, 55.9, 43.7.



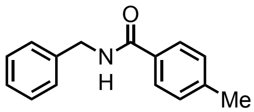
***N*-benzyl-2-bromoxybenzamide (3d)[3]**

Substrate **3d** was obtained as white powder (34.8 mg, 60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (td, *J* = 7.8, 7.7, 1.5 Hz, 2H), 7.43 – 7.24 (m, 7H), 6.25 (s, 1H), 4.66 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 137.6, 137.6, 133.4, 131.3, 129.6, 128.8, 128.0, 127.7, 127.6, 119.3, 44.3.



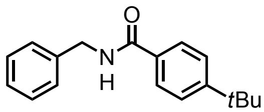
N-benzyl-3-methoxybenzamide (3e)[4]

Substrate **3e** was obtained as white powder (41.0 mg, 85%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.35 (m, 1H), 7.33 – 7.19 (m, 7H), 7.03 (brs, 1H), 6.99 – 6.94 (m, 1H), 4.53 (d, *J*= 5.6 Hz, 2H), 3.74 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 159.6, 138.2, 135.7, 129.3, 128.5, 127.6, 127.2, 118.8, 117.5, 112.3, 55.2, 43.8.



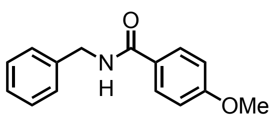
N-benzyl-4-methylbenzamide (3f)[5]

Substrate **3f** was obtained as white powder (35.6 mg, 79%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.39 – 7.24 (m, 5H), 7.21 (d, *J* = 7.9 Hz, 2H), 6.46 (s, 1H), 4.62 (d, *J* = 5.6 Hz, 2H), 2.38 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 141.9, 138.3, 131.5, 129.2, 128.7, 127.9, 127.5, 126.9, 44.0, 21.4.



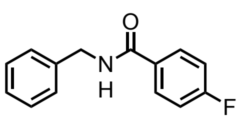
N-benzyl-4-(*tert*-butyl)benzamide (3g)[6]

Substrate **3g** was obtained as white powder (41.7 mg, 78%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.24 (m, 5H), 6.50 (s, 1H), 4.63 (d, *J* = 5.7 Hz, 2H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 155.0, 138.4, 131.5, 128.7, 127.8, 127.5, 126.8, 125.5, 44.0, 34.9, 31.1.



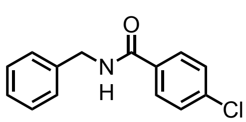
N-benzyl-4-methoxybenzamide (3h)[1]

Substrate **3h** was obtained as white powder (40.5 mg, 84%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.7 Hz, 2H), 7.40 – 7.24 (m, 5H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.37 (s, 1H), 4.62 (d, *J* = 5.7 Hz, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 162.2, 138.4, 128.7, 127.9, 127.5, 126.7, 113.7, 55.4, 44.1.



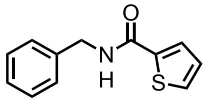
N-benzyl-4-fluorobenzamide (3i)[7]

Substrate **3i** was obtained as white powder (34.3 mg, 75%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.74 (m, 2H), 7.40 – 7.24 (m, 5H), 7.17 – 6.99 (m, 2H), 6.41 (s, 1H), 4.62 (d, *J* = 5.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.26, 164.7 (d, *J* = 252.0 Hz), 138.0, 130.5, 129.3 (d, *J* = 8.9 Hz), 128.8, 127.9, 127.7, 115.6 (d, *J* = 21.9 Hz), 44.2.



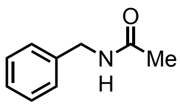
N-benzyl-4-chlorobenzamide (3j)[7]

Substrate **3j** was obtained as white powder (37.8 mg, 77%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 8.6 Hz, 2H), 7.39 (d, J = 8.6 Hz, 2H), 7.37 – 7.27 (m, 5H), 6.42 (s, 1H), 4.62 (d, J = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 137.9, 137.8, 132.7, 128.8, 128.4, 127.9, 127.7, 44.2.



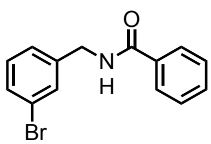
***N*-benzylthiophene-2-carboxamide (3k)[8]**

Substrate **3k** was obtained as white powder (34.8 mg, 80%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (dd, J = 3.7, 1.1 Hz, 1H), 7.47 (dd, J = 5.0, 1.1 Hz, 1H), 7.38 – 7.26 (m, 5H), 7.08 – 7.03 (m, 1H), 6.39 (s, 1H), 4.61 (d, J = 5.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.8, 138.7, 138.0, 130.0, 128.7, 128.1, 127.9, 127.6, 126.6, 44.0.



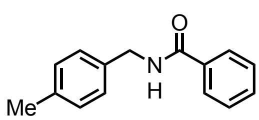
***N*-benzylacetamide (3l)[9]**

Substrate **3l** was obtained as white powder (7.5 mg, 25%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.23 (m, 5H), 5.82 (s, 1H), 4.42 (d, J = 5.7 Hz, 2H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 138.2, 128.7, 127.8, 127.5, 43.8, 23.3.



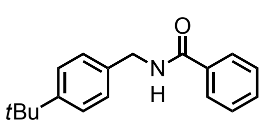
***N*-(3-bromobenzyl)benzamide (4a)[10]**

Substrate **4a** was obtained as white powder (43.5 mg, 75%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.74 (m, 2H), 7.55 – 7.45 (m, 2H), 7.45 – 7.36 (m, 3H), 7.31 – 7.23 (m, 1H), 7.19 (t, J = 7.7 Hz, 1H), 6.63 (s, 1H), 4.59 (d, J = 5.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 140.6, 134.1 131.7, 130.7, 130.6, 130.3, 128.6, 127.0, 126.4, 122.7, 43.4.



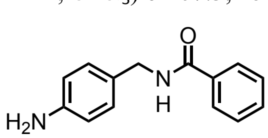
***N*-(4-methylbenzyl)benzamide (4b)[8]**

Substrate **4b** was obtained as white powder (40.6 mg, 90%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.74 (m, 2H), 7.52 – 7.45 (m, 1H), 7.42 – 7.38 (m, 2H), 7.27 – 7.21 (m, 2H), 7.15 (d, J = 7.8 Hz, 2H), 6.45 (s, 1H), 4.59 (d, J = 5.5 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 137.3, 135.1, 134.4, 131.4, 129.4, 128.5, 127.9, 126.9, 43.9, 21.1.



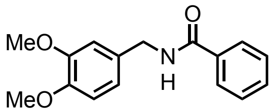
***N*-(4-(*tert*-butyl)benzyl)benzamide (4c)[11]**

Substrate **4c** was obtained as white powder (48.7 mg, 91%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.75 (m, 2H), 7.52 – 7.45 (m, 1H), 7.45 – 7.33 (m, 4H), 7.29 (d, J = 8.2 Hz, 2H), 6.48 (s, 1H), 4.60 (d, J = 5.5 Hz, 2H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 150.6, 135.1, 134.5, 131.4, 128.5, 127.7, 126.9, 125.7, 43.8, 34.5, 31.3.



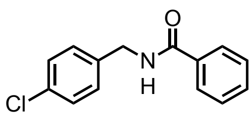
***N*-(4-aminobenzyl)benzamide (4d)[12]**

Substrate **4d** was obtained as gray powder (36.7 mg, 86%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.72 (m, 2H), 7.51 – 7.43 (m, 1H), 7.39 (t, *J* = 7.4 Hz, 2H), 7.13 (d, *J* = 8.3 Hz, 2H), 6.64 (d, *J* = 8.4 Hz, 2H), 6.40 (s, 1H), 4.50 (d, *J* = 5.4 Hz, 2H), 3.57 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 145.9, 134.5, 131.3, 129.2, 128.5, 127.9, 126.9, 115.2, 43.8.



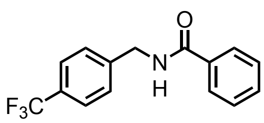
***N*-(3,4-dimethoxybenzyl)benzamide (4e)**

Substrate **4e** was obtained as white powder (44.0 mg, 81%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.4 Hz, 2H), 7.50 – 7.40 (m, 1H), 7.40 – 7.31 (m, 2H), 7.03 (s, 1H), 6.84 (d, *J* = 6.1 Hz, 2H), 6.76 (dd, *J* = 8.6, 1.9 Hz, 1H), 4.51 (d, *J* = 5.6 Hz, 2H), 3.80 (s, 3H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 148.9, 148.2, 134.2, 131.2, 130.8, 128.3, 126.8, 119.9, 111.1, 111.1, 55.7, 55.6, 43.6; HRMS m/z (ESI) calcd for $C_{16}H_{18}NO_3$ $(M + H)^+$ 272.1281, found 272.1286.



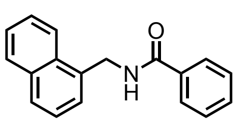
***N*-(4-chlorobenzyl)benzamide (4f)[8]**

Substrate **4f** was obtained as white powder (39.3 mg, 80%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.6 Hz, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.34 – 7.21 (m, 4H), 6.61 (s, 1H), 4.58 (d, *J* = 5.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 136.8, 134.1, 133.3, 131.6, 129.1, 128.8, 128.6, 126.9, 43.3.



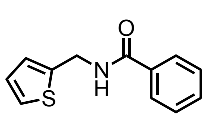
***N*-(4-trifluoromethylbenzyl)benzamide (4g)[8]**

Substrate **4g** was obtained as white powder (44.7 mg, 80%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.6 Hz, 2H), 7.58 (d, *J* = 7.9 Hz, 2H), 7.51 (t, *J* = 7.4 Hz, 1H), 7.48 – 7.39 (m, 4H), 6.67 (s, 1H), 4.68 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 142.4, 134.0, 131.8, 129.8 (q, *J* = 32.8, 32.0 Hz), 128.6, 127.9, 127.0, 125.6 (q, *J* = 3.6 Hz), 124.0 (q, *J* = 272.4 Hz), 43.5.



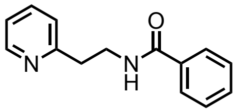
***N*-(naphthalen-1-ylmethyl)benzamide (4h)[10]**

Substrate **4h** was obtained as white powder (41.8 mg, 80%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 – 8.04 (m, 1H), 7.89 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.87 – 7.82 (m, 1H), 7.80 – 7.71 (m, 2H), 7.58 – 7.34 (m, 7H), 6.37 (s, 1H), 5.09 (d, *J* = 5.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 134.3, 133.9, 133.4, 131.5, 131.5, 128.8, 128.8, 128.5, 126.9, 126.8, 126.0, 125.4, 123.5, 42.4.



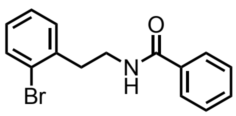
***N*-(thiophen-2-ylmethyl)benzamide (4i)[8]**

Substrate **4i** was obtained as white powder (26.1 mg, 60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.73 (m, 2H), 7.50 – 7.42 (m, 1H), 7.41 – 7.33 (m, 2H), 7.20 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.99 (dt, *J* = 3.2, 1.0, 1.0 Hz, 1H), 6.93 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.85 (s, 1H), 4.76 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 140.8, 134.0, 131.5, 128.4, 127.0, 126.8, 126.0, 125.1, 38.7.



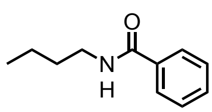
N-(2-(pyridin-2-yl)ethyl)benzamide (4 j)[13]

Substrate **4j** was obtained as white powder (38.0 mg, 84%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 3.1 Hz, 1H), 7.78 (d, *J* = 7.2 Hz, 2H), 7.72 (s, 1H), 7.60 (td, *J* = 7.7, 7.6, 1.9 Hz, 1H), 7.44 (m, 1H), 7.38 (dd, *J* = 8.2, 6.5 Hz, 2H), 7.16 (m, 2H), 3.83 (q, *J* = 6.1, 6.1, 6.1 Hz, 2H), 3.08 (t, *J* = 6.3, 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.2, 159.6, 148.9, 136.6, 134.5, 131.1, 128.3, 126.8, 123.4, 121.5, 39.2, 36.5.



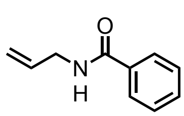
N-(2-bromophenethyl)benzamide (4k)[14]

Substrate **4k** was obtained as white powder (48.0 mg, 79%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 3.1 Hz, 1H), 7.78 (d, *J* = 7.2 Hz, 2H), 7.72 (s, 1H), 7.60 (td, *J* = 7.7, 7.6, 1.9 Hz, 1H), 7.49 – 7.41 (m, 1H), 7.38 (dd, *J* = 8.2, 6.5 Hz, 2H), 7.23 – 7.10 (m, 2H), 3.83 (q, *J* = 6.1, 6.1, 6.1 Hz, 2H), 3.08 (t, *J* = 6.3, 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 138.3, 134.5, 133.0, 131.4, 131.1, 128.5, 128.3, 127.7, 126.8, 124.6, 39.8, 35.7.



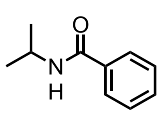
_N_-butylbenzamide (4l)[15]

Substrate **4l** was obtained as white powder (30.4 mg, 86%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.72 (m, 2H), 7.52 – 7.43 (m, 1H), 7.43 – 7.36 (m, 2H), 6.35 (s, 1H), 3.51 – 3.38 (m, 2H), 1.65 – 1.54 (m, 2H), 1.45 – 1.35 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 134.8, 131.2, 128.4, 126.8, 39.8, 31.7, 20.1, 13.7.



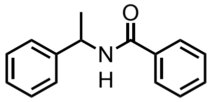
_N_-allylbenzamide (4m)[16]

Substrate **4m** was obtained as white powder (27.7 mg, 86%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.75 (m, 2H), 7.52 – 7.45 (m, 1H), 7.44 – 7.35 (m, 2H), 6.69 (s, 1H), 5.91 (ddt, *J* = 17.2, 10.2, 5.6, 5.6 Hz, 1H), 5.30 – 5.11 (m, 2H), 4.05 (tt, *J* = 5.7, 5.7, 1.6, 1.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.4, 134.3, 134.1, 131.3, 128.4, 126.9, 116.4, 42.3.



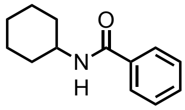
_N_-isopropylbenzamide (4n)[15]

Substrate **4n** was obtained as white powder (23.5 mg, 72%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 – 7.71 (m, 2H), 7.52 – 7.45 (m, 1H), 7.41 (t, *J* = 7.3 Hz, 2H), 5.95 (s, 1H), 4.42 – 4.20 (m, 1H), 1.26 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 135.0, 131.2, 128.5, 126.8, 41.9, 22.8.



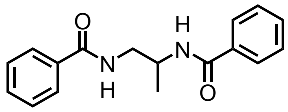
N-(1-phenylethyl)benzamide (4o)[15]

Substrate **4o** was obtained as white powder (27.0 mg, 60%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.3 Hz, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.42 – 7.32 (m, 6H), 7.31 – 7.23 (m, 1H), 6.40 (s, 1H), 5.37 – 5.28 (m, 1H), 1.60 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 143.1, 134.6, 131.4, 128.7, 128.5, 127.4, 126.9, 126.2, 49.2, 21.7.



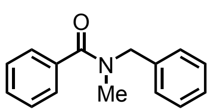
N-cyclohexylbenzamide (4p)[15]

Substrate **4p** was obtained as white powder (28.9 mg, 71%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.8 Hz, 2H), 7.48 (t, *J* = 7.0 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 5.98 (s, 1H), 4.06 – 3.92 (m, 1H), 2.06 – 2.01 (m, 2H), 1.79 – 1.72 (m, 2H), 1.67 – 1.63 (m, 1H), 1.56 – 1.36 (m, 2H), 1.37 – 1.11 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 135.1, 131.2, 128.5, 126.8, 48.6, 33.2, 25.6, 24.9.



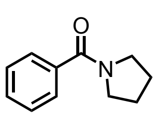
N,N'-(propane-1,2-diyl)dibenzamide (4q)[17]

Substrate **4q** was obtained as white powder (31.1 mg, 55%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.76 (m, 4H), 7.52 – 7.44 (m, 2H), 7.44 – 7.32 (m, 5H), 7.12 (d, *J* = 7.3 Hz, 1H), 4.42 – 4.33 (m, 1H), 3.74 – 3.65 (m, 1H), 3.50 – 3.45 (m, 1H), 1.31 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 168.2, 134.0, 133.9, 131.6, 131.5, 128.5, 128.5, 127.01, 127.00, 47.2, 46.5, 18.4.



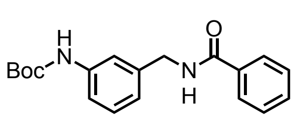
N-benzyl-N-methylbenzamide (4s)[18] (rotamer)

Substrate **4s** was obtained as colorless oil (32.8 mg, 73%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.25 (m, 9H), 7.16 (s, 1H), 4.76 (s, 1H), 4.51 (s, 1H), 3.03 (s, 1.5H), 2.85 (s, 1.5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 171.5, 136.9, 136.5, 136.1, 132.7, 129.8, 129.5, 128.6, 128.3, 128.1, 128.1, 127.5, 126.9, 126.7, 55.1, 50.7, 36.9, 33.1.



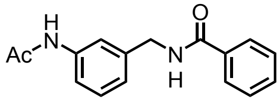
phenyl(pyrrolidin-1-yl)methanone (4t)[18]

Substrate **4t** was obtained as colorless oil (19.3 mg, 55%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.47 (m, 2H), 7.43 – 7.35 (m, 3H), 3.64 (t, 7.0 Hz, 2H), 3.42 (t, 6.6 Hz, 2H), 1.99 – 1.81 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 137.1, 129.6, 128.1, 126.9, 49.4, 46.0, 26.2, 24.3.



tert-butyl (3-(benzamidomethyl)phenyl)carbamate (5a)

Substrate **5a** was obtained as white powder (56.1 mg, 86%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.76 (m, 2H), 7.52 – 7.46 (m, 1H), 7.45 – 7.36 (m, 3H), 7.27 (dd, *J* = 10.9, 4.9 Hz, 2H), 7.06 – 6.99 (m, 1H), 6.63 (s, 1H), 6.58 (s, 1H), 4.58 (d, *J* = 5.7 Hz, 2H), 1.50 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.36, 152.76, 139.13, 138.76, 134.29, 131.51, 129.37, 128.54, 126.98, 122.48, 117.97, 117.75, 80.60, 44.02, 28.29; HRMS m/z (ESI) calcd for $C_{19}H_{23}N_2O_3$ $(M + H)^+$ 327.1703, found 327.1709.



***N*-(3-acetamidobenzyl)benzamide (5b)**

Substrate **5b** was obtained as white powder (45.5 mg, 84%); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.82 – 7.74 (m, 2H), 7.51 – 7.32 (m, 5H), 7.23 – 7.14 (m, 1H), 7.08 – 6.96 (m, 2H), 4.48 (s, 2H), 2.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2, 167.8, 138.9, 138.5, 134.0, 131.5, 129.1, 128.5, 127.0, 123.1, 119.2, 119.2, 43.7, 24.2; HRMS m/z (ESI) calcd for $C_{16}H_{17}N_2O_2$ $(M + H)^+$ 269.1285, found 269.1289.

Reference：

(1) B. Roberts, D. Liptrot, L. Alcaraz, T. Luker and M. J. Stocks, *Org. Lett.*, 2010, **12**, 4280-4283.

(2) J. R. Martinelli, D. A. Watson, D. M. M. Freckmann, T. E. Barder and S. L. Buchwald, *J. Org. Chem.*, 2008, **73**, 7102-7107.

(3) P. Thansandote, D. G. Hulcoop, M. Langer and M. Lautens, *J. Org. Chem.*, 2009, **74**, 1673-1678

(4) W. Ren and M. Yamane, *J. Org. Chem.*, 2010, **75**, 3017-3020.

(5) Y. Q. Wan, M. Alterman, M. Larhed and A. Hallberg, *J. Org. Chem.*, 2002, **67**, 6232-6235.

(6) N. Iqbal and E. J. Cho, *J. Org. Chem.*, 2016, **81**, 1905-1911.

(7) R. A. Green, D. Pletcher, S. G. Leach and R. C. D. Brown, *Org. Lett.*, 2016, **18**, 1198-1201.

(8) N. Wang, X. Zou, J. Ma and F. Li, *Chem. Commun.*, 2014, **50**, 8303-8305.

(9) J. E. Taylor, M. D. Jones, J. M. J. Williams and S. D. Bull, *J. Org. Chem.*, 2012, **77**, 2808-2818.

(10) S. Kerdphon, X. Quan, V. S. Parihar and P. G. Andersson, *J. Org. Chem.*, 2015, **80**, 11529-11537.

(11) L. Rubio-Perez, P. Sharma, F. Javier Perez-Flores, L. Velasco, J. Luis Arias and A. Cabrera, *Tetrahedron*, 2012, **68**, 2342-2348.

(12) C. Baumgartner, C. Eberle, F. Diederich, S. Lauw, F. Rohdich, W. Eisenreich and A. Barber, *Helv. Chim. Acta*, 2007, **90**, 1043-1068.

(13) S. Inoue, H. Shiota, Y. Fukumoto and N. Chatani, *J. Am. Chem. Soc.*, 2009, **131**, 6898-6899.

(14) S. Balieu, K. Toutah, L. Carro, L.-M. Chamoreau, H. Rousseliere and C. Courillon, *Tetrahedron Lett.*, 2011, **52**, 2876-2880.

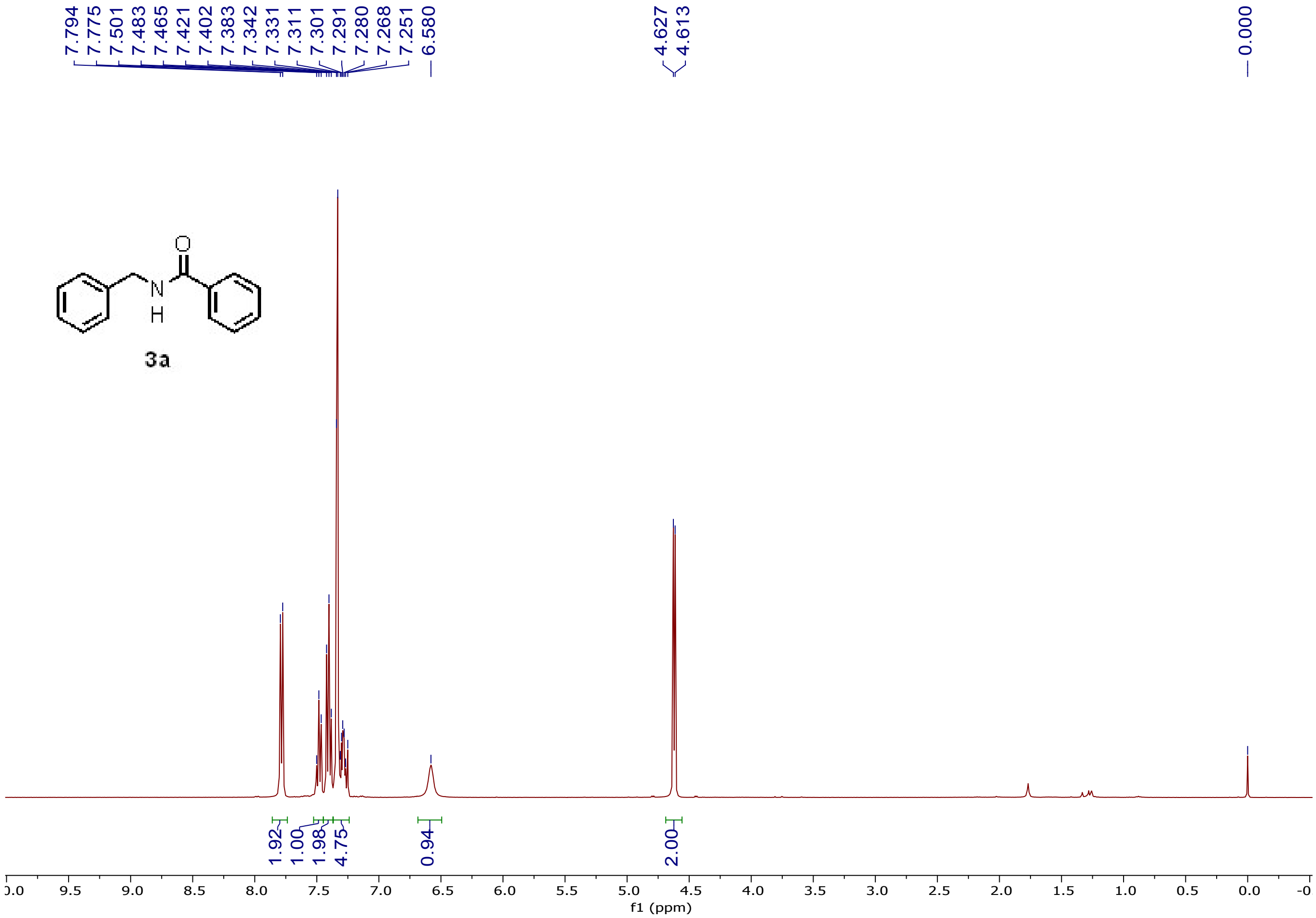
(15) X. Bantreil, N. Kanfar, N. Gehin, E. Golliard, P. Ohlmann, J. Martinez and F. Lamaty, *Tetrahedron*, 2014, **70**, 5093-5099.

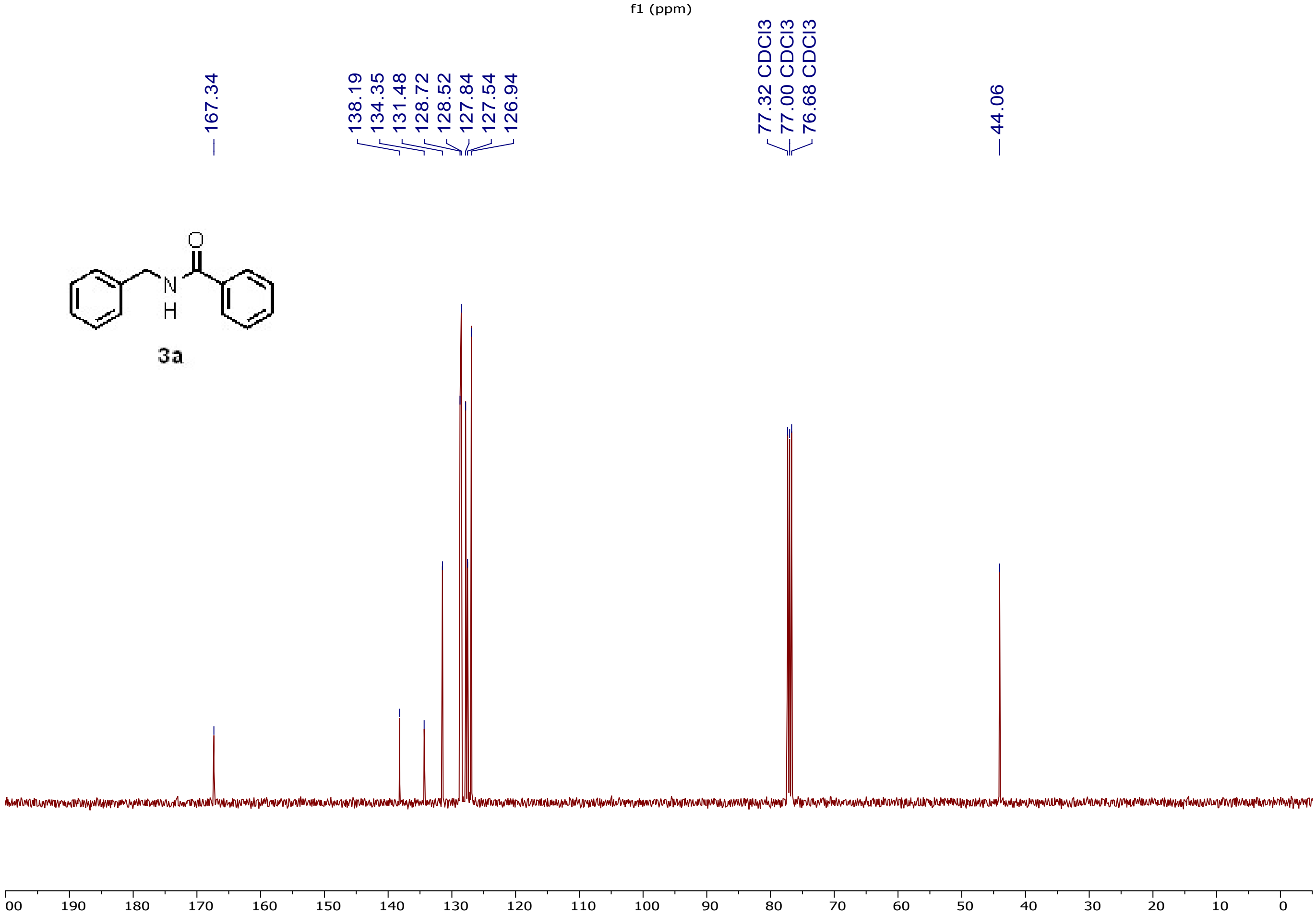
(16) D. S. Hemming, E. P. Talbot and P. G. Steel, *Tetrahedron Lett.*, 2017, **58**, 17-20.

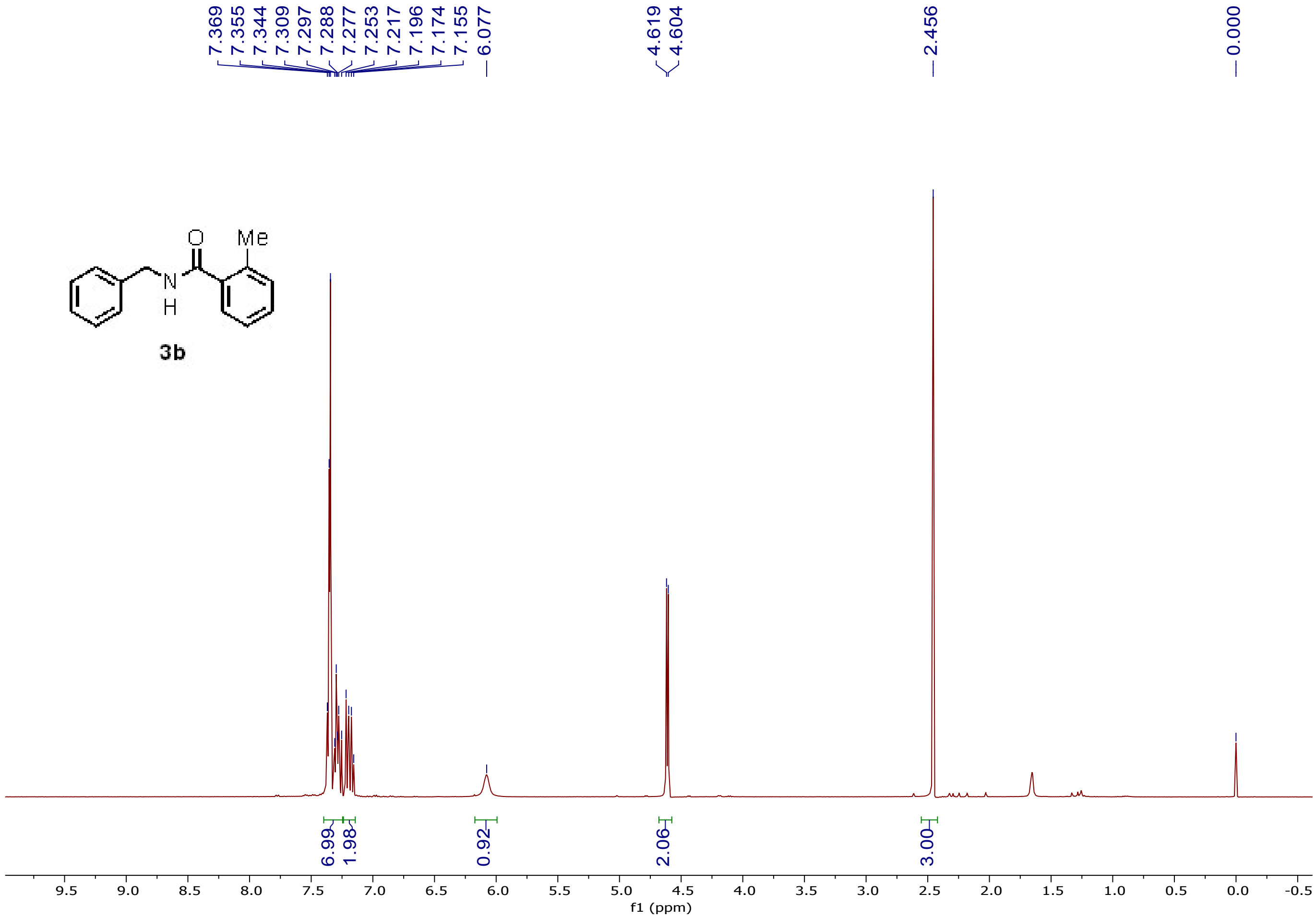
(17) R. Kuwano, N. Kameyama and R. Ikeda, *J. Am. Chem. Soc.*, 2011, **133**, 7312-7315.

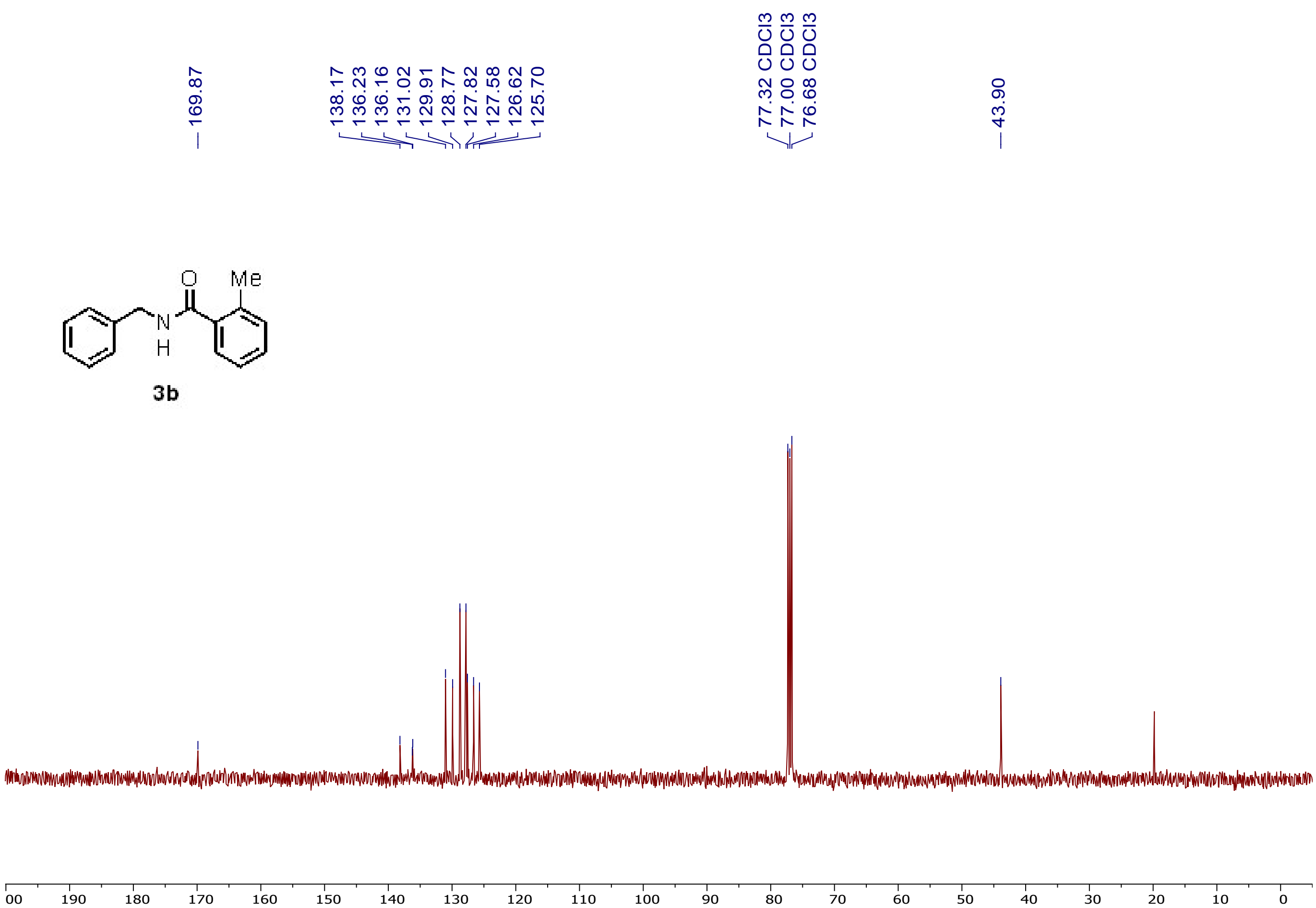
(18) Y. Liu, S. Shi, M. Achtenhagen, R. Liu and M. Szostak, *Org. Lett.*, 2017, **19**, 1614-1617.

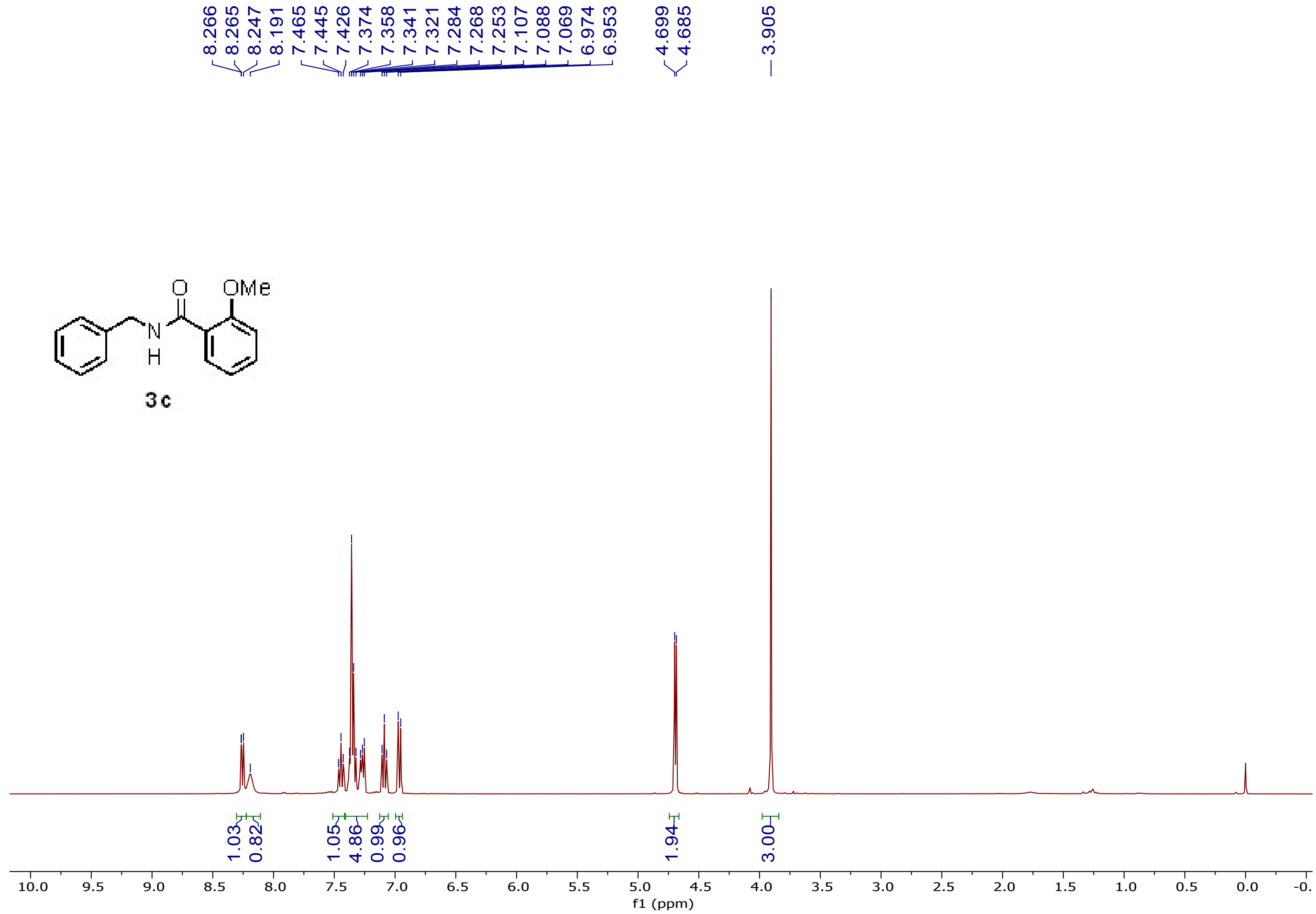
3. ^{1}H and ^{13}C NMR Spectra

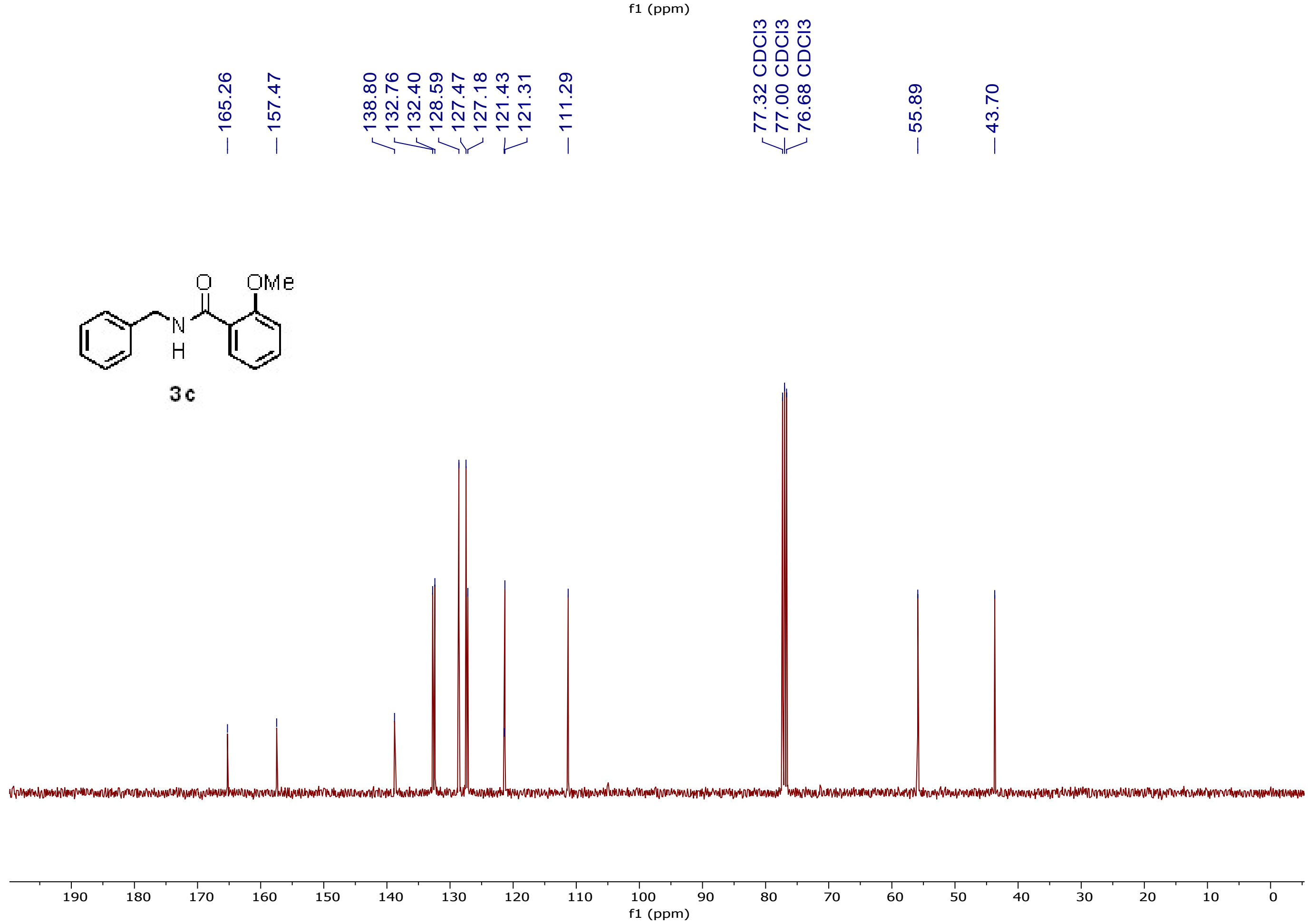


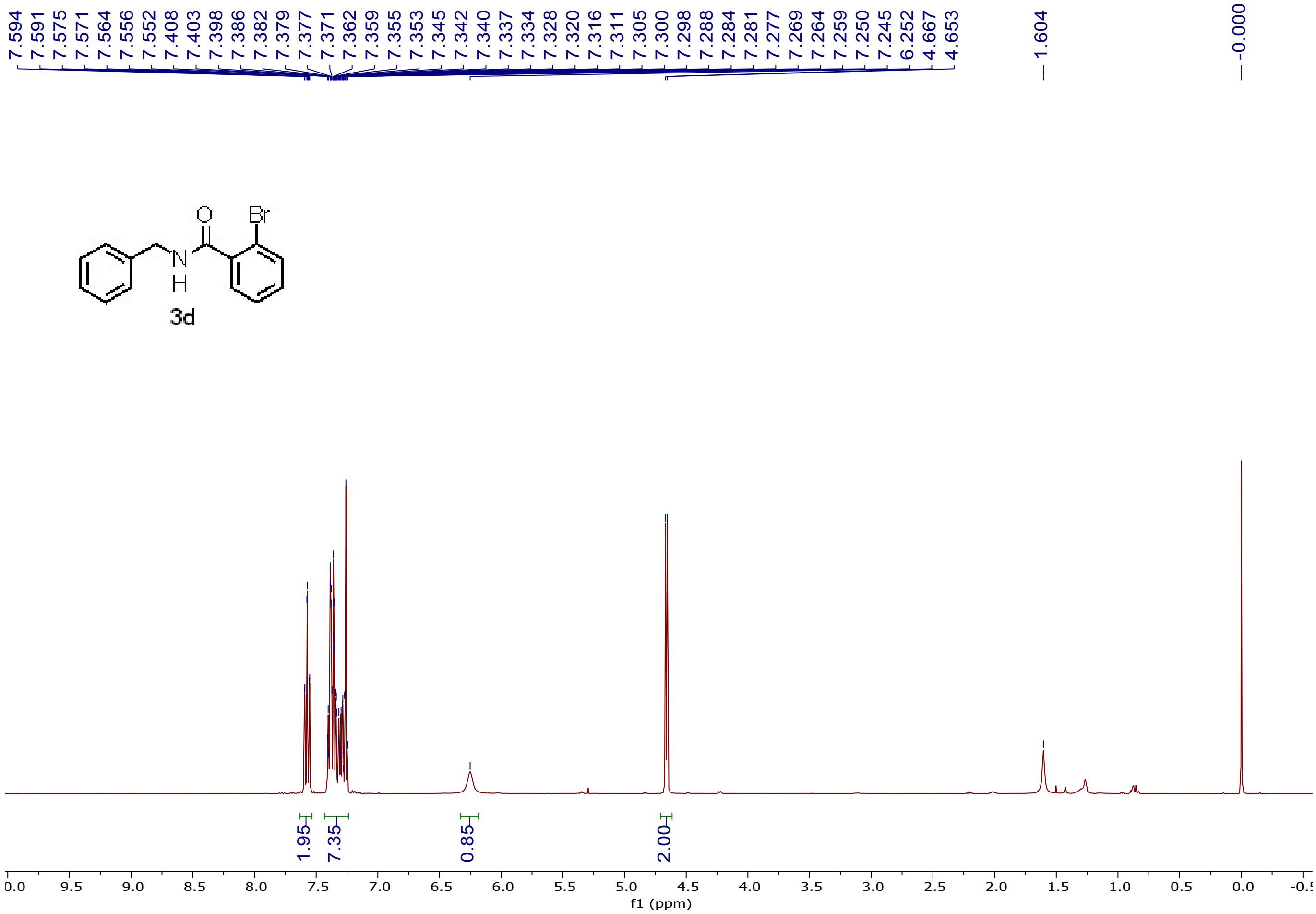


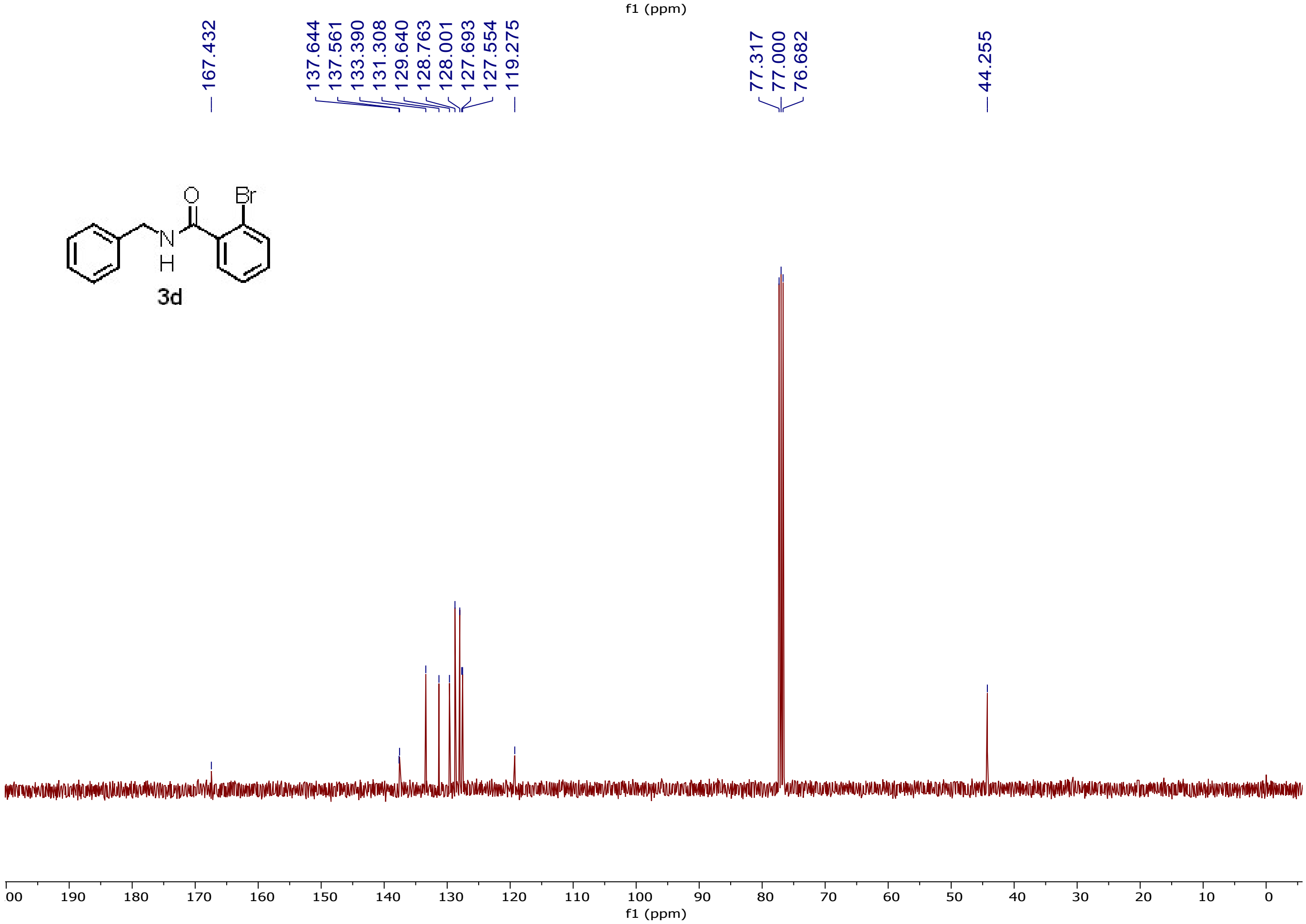

7.369
7.355
7.344
7.309
7.297
7.288
7.277
7.253
7.217
7.196
7.174
7.155
6.077
4.619
4.604
2.456
0.000
O
Me
N
H
3b
6.99
1.98
0.92
2.06
3.00
f1 (ppm)

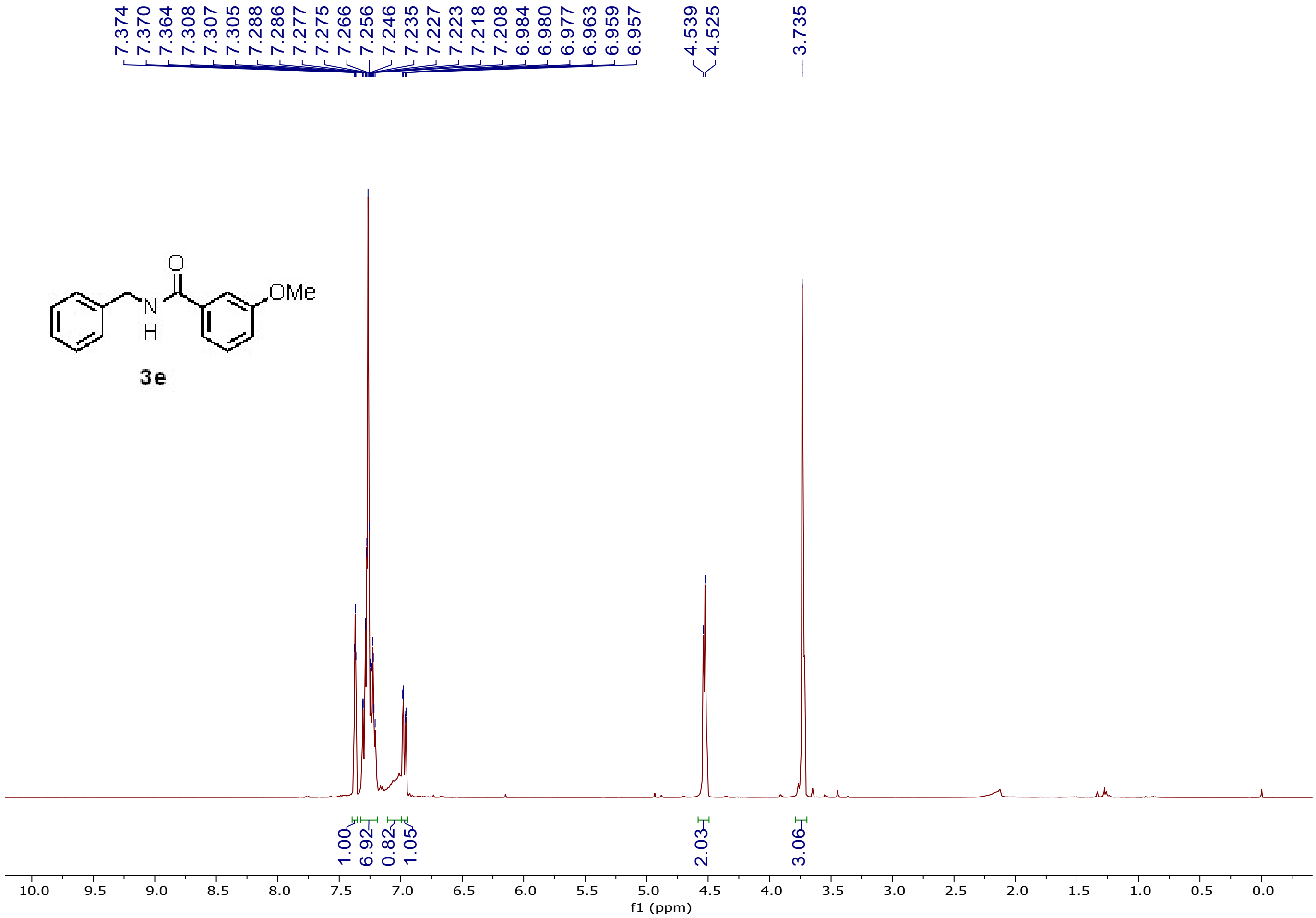

169.87
138.17
136.23
136.16
131.02
129.91
128.77
127.82
127.58
126.62
125.70
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
43.90
O
Me
N
H
3b
f1 (ppm)

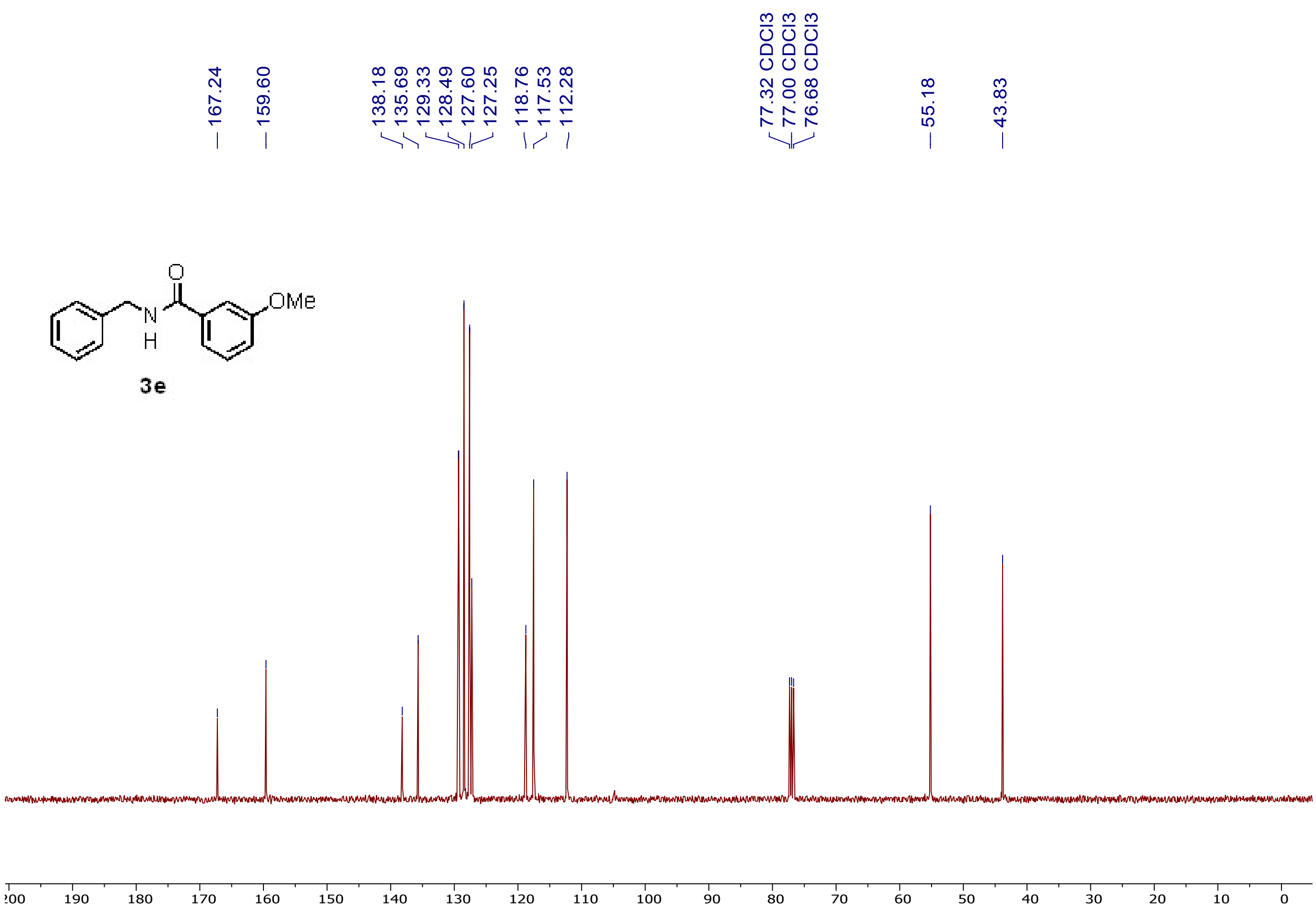

8.266
8.265
8.247
8.191
7.465
7.445
7.426
7.374
7.358
7.341
7.321
7.284
7.268
7.253
7.107
7.088
7.069
6.974
6.953
4.699
4.685
3.905
OMe
O
N
H
3c
1.03
0.82
1.05
4.86
0.99
0.96
1.94
3.00
f1 (ppm)

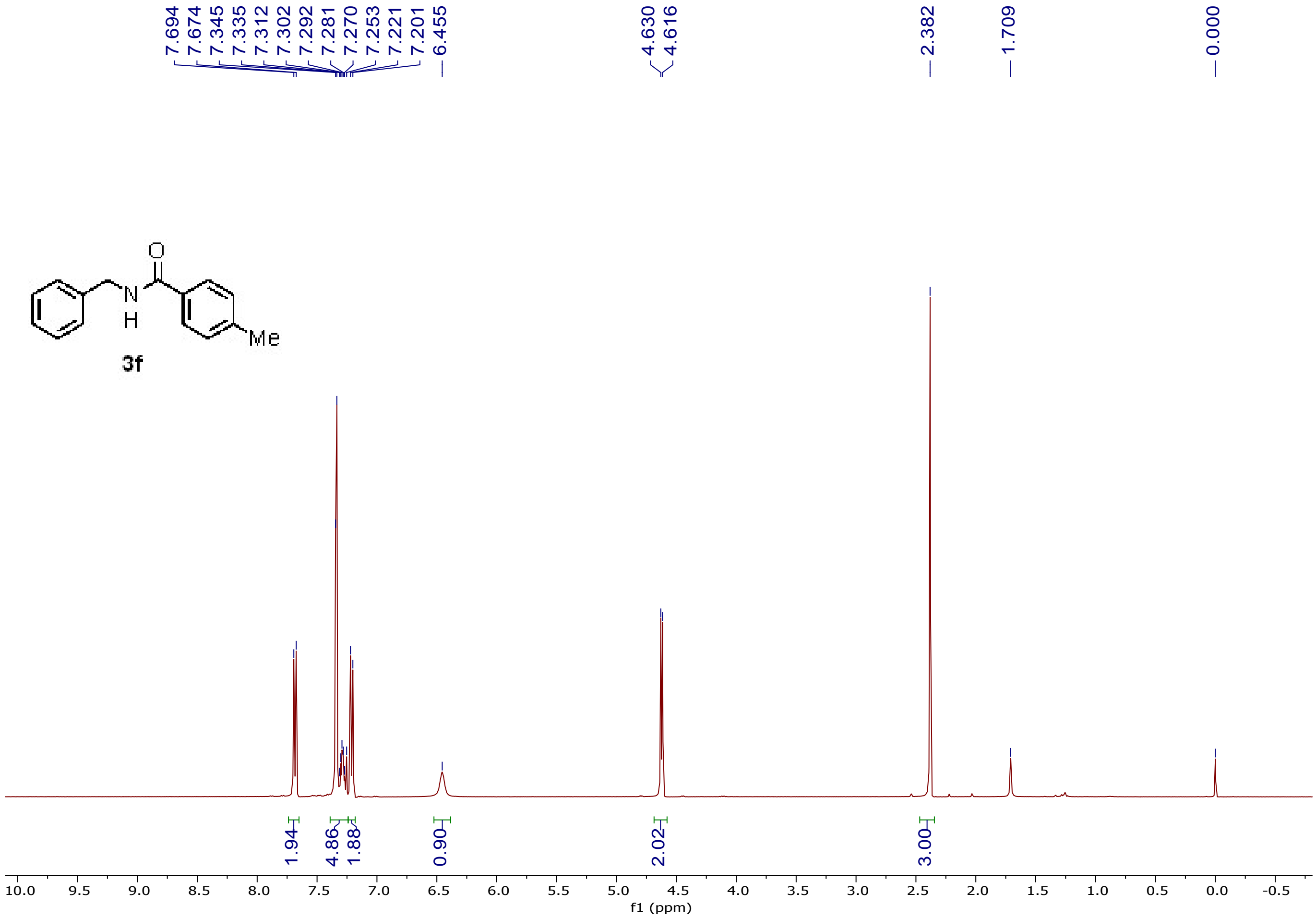

165.26
157.47
138.80
132.76
132.40
128.59
127.47
127.18
121.43
121.31
111.29
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
55.89
43.70
OMe
O
N
H
3c
f1 (ppm)

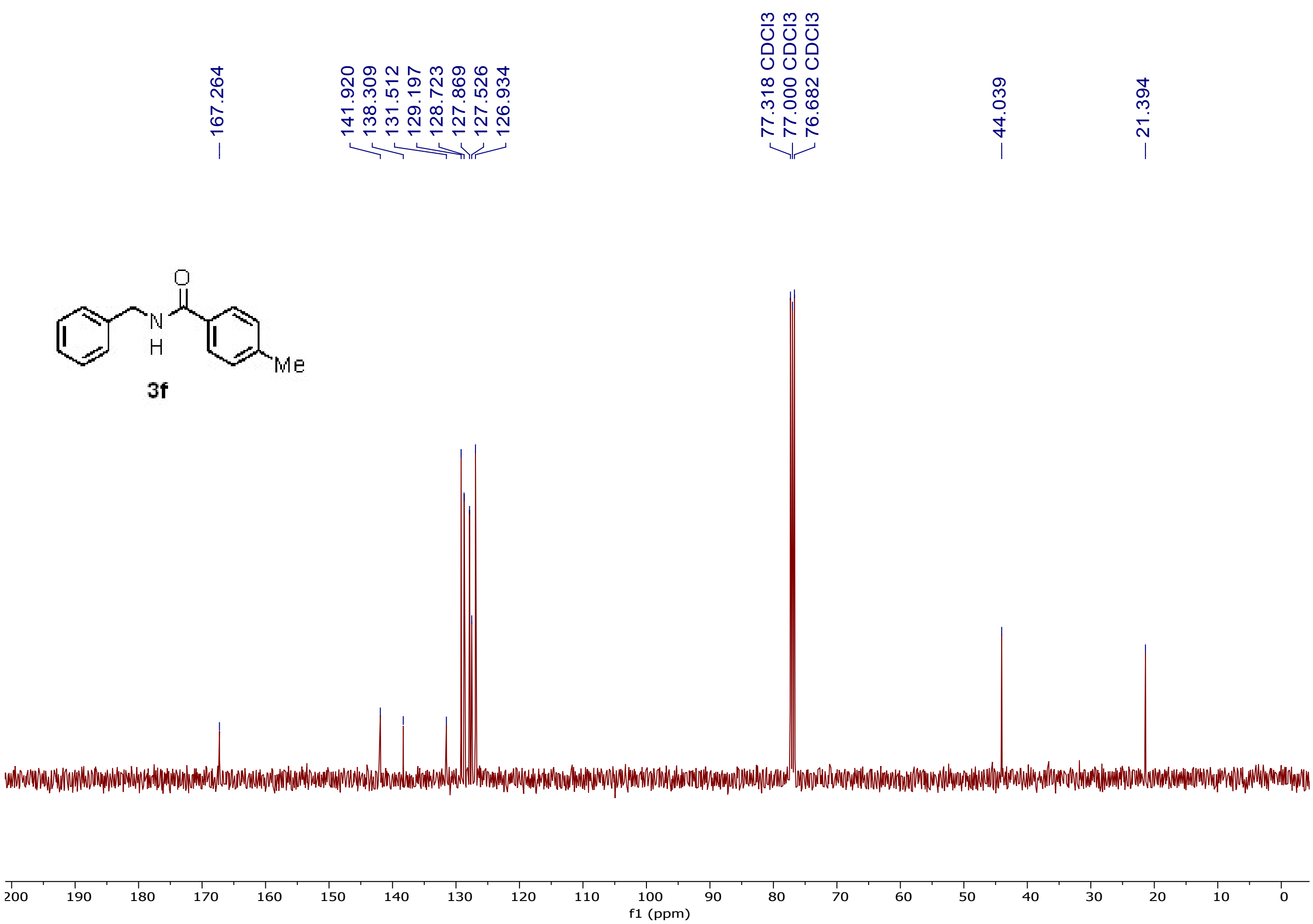

7.594
7.591
7.575
7.571
7.564
7.556
7.552
7.408
7.403
7.398
7.386
7.382
7.379
7.377
7.371
7.362
7.359
7.355
7.353
7.345
7.342
7.340
7.337
7.334
7.328
7.320
7.316
7.311
7.305
7.300
7.298
7.288
7.284
7.281
7.277
7.269
7.264
7.259
7.250
7.245
6.252
4.667
4.653
1.604
-0.000
Br
O
N
H
3d
1.95
7.35
0.85
2.00
f1 (ppm)

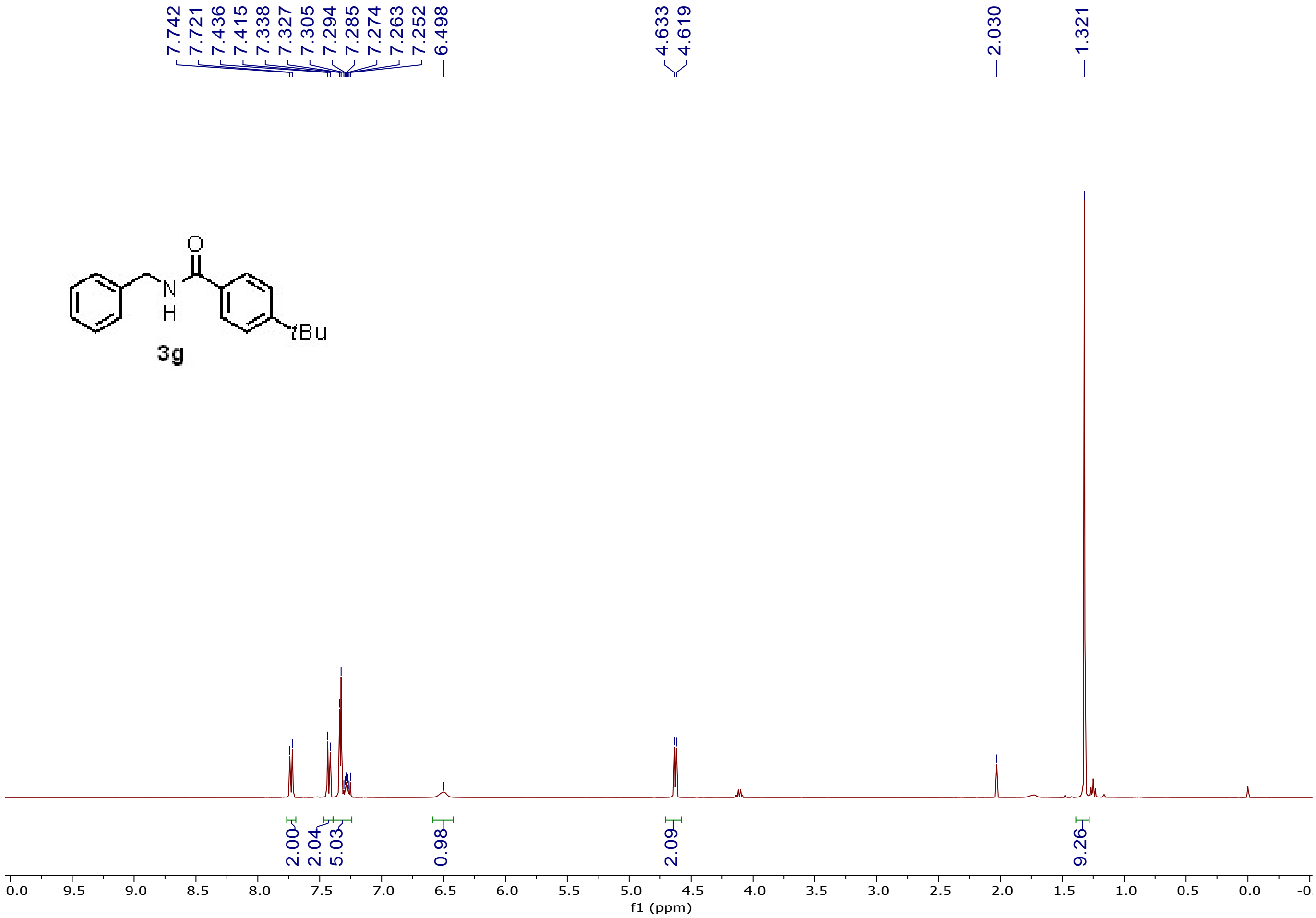

167.432
137.644
137.561
133.390
131.308
129.640
128.763
128.001
127.693
127.554
119.275
77.317
77.000
76.682
44.255
Br
O
N
H
3d
f1 (ppm)

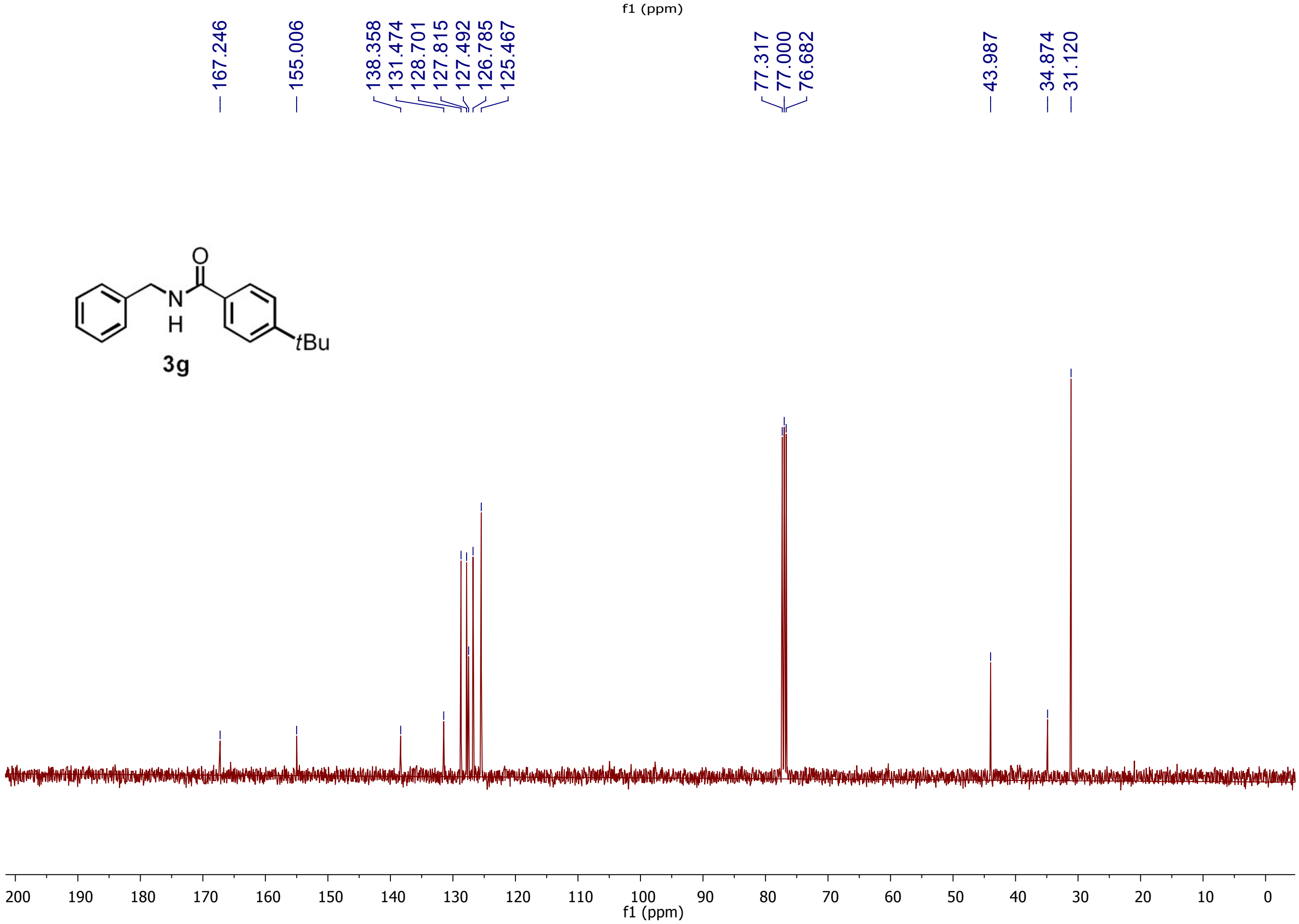

7.374
7.370
7.364
7.308
7.307
7.305
7.288
7.286
7.277
7.275
7.266
7.256
7.246
7.235
7.227
7.223
7.218
7.208
6.984
6.980
6.977
6.963
6.959
6.957
4.539
4.525
3.735
OMe
3e
1.00
6.92
0.82
1.05
2.03
3.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

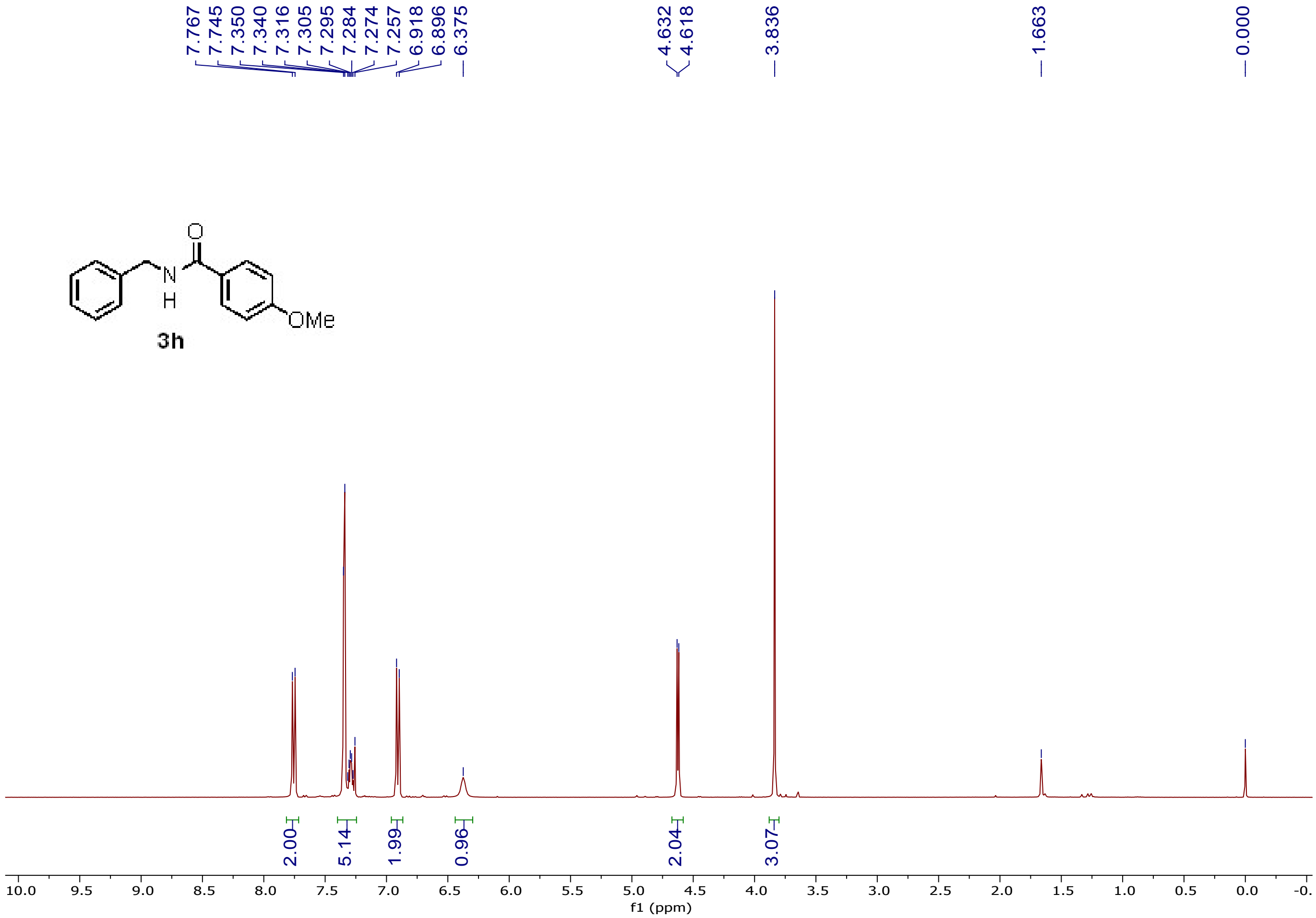

167.24
159.60
138.18
135.69
129.33
128.49
127.60
127.25
118.76
117.53
112.28
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
55.18
43.83
OMe
3e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

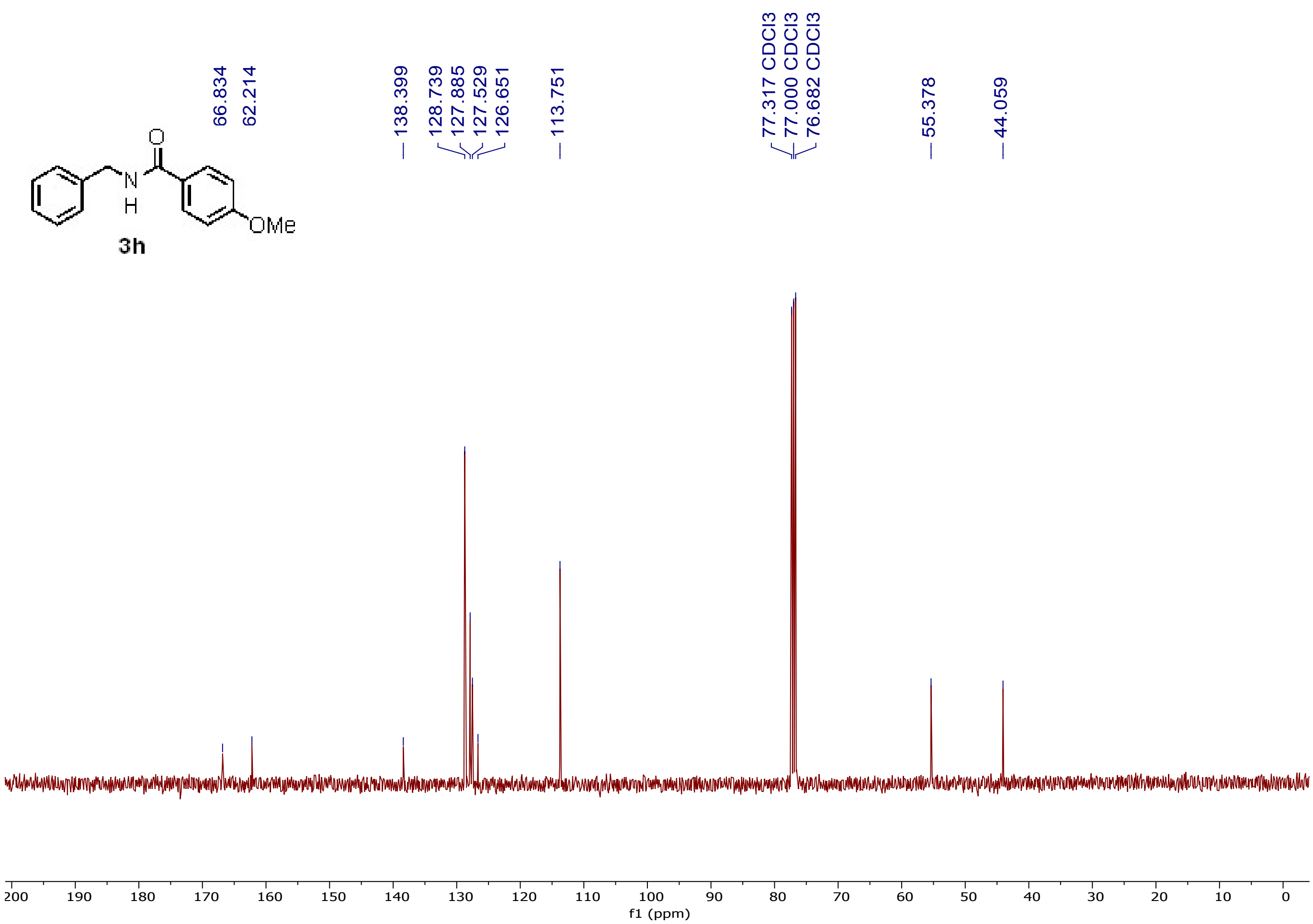

7.694
7.674
7.345
7.335
7.312
7.302
7.292
7.281
7.270
7.253
7.221
7.201
6.455
4.630
4.616
2.382
1.709
0.000
O
N
H
Me
3f
1.94
4.86
1.88
0.90
2.02
3.00
f1 (ppm)

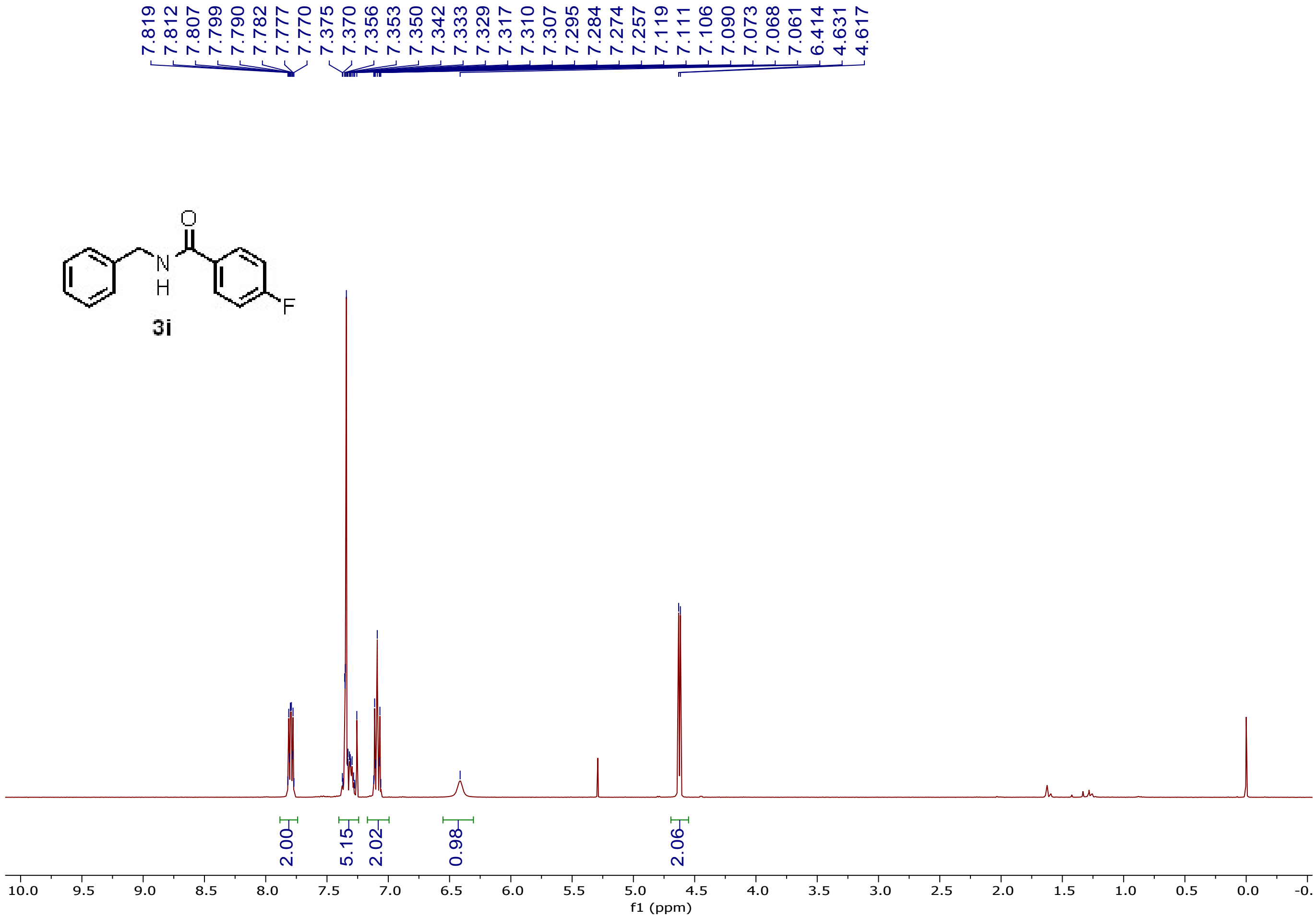

167.264
141.920
138.309
131.512
129.197
128.723
127.869
127.526
126.934
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
44.039
21.394
O
N
H
Me
3f
f1 (ppm)

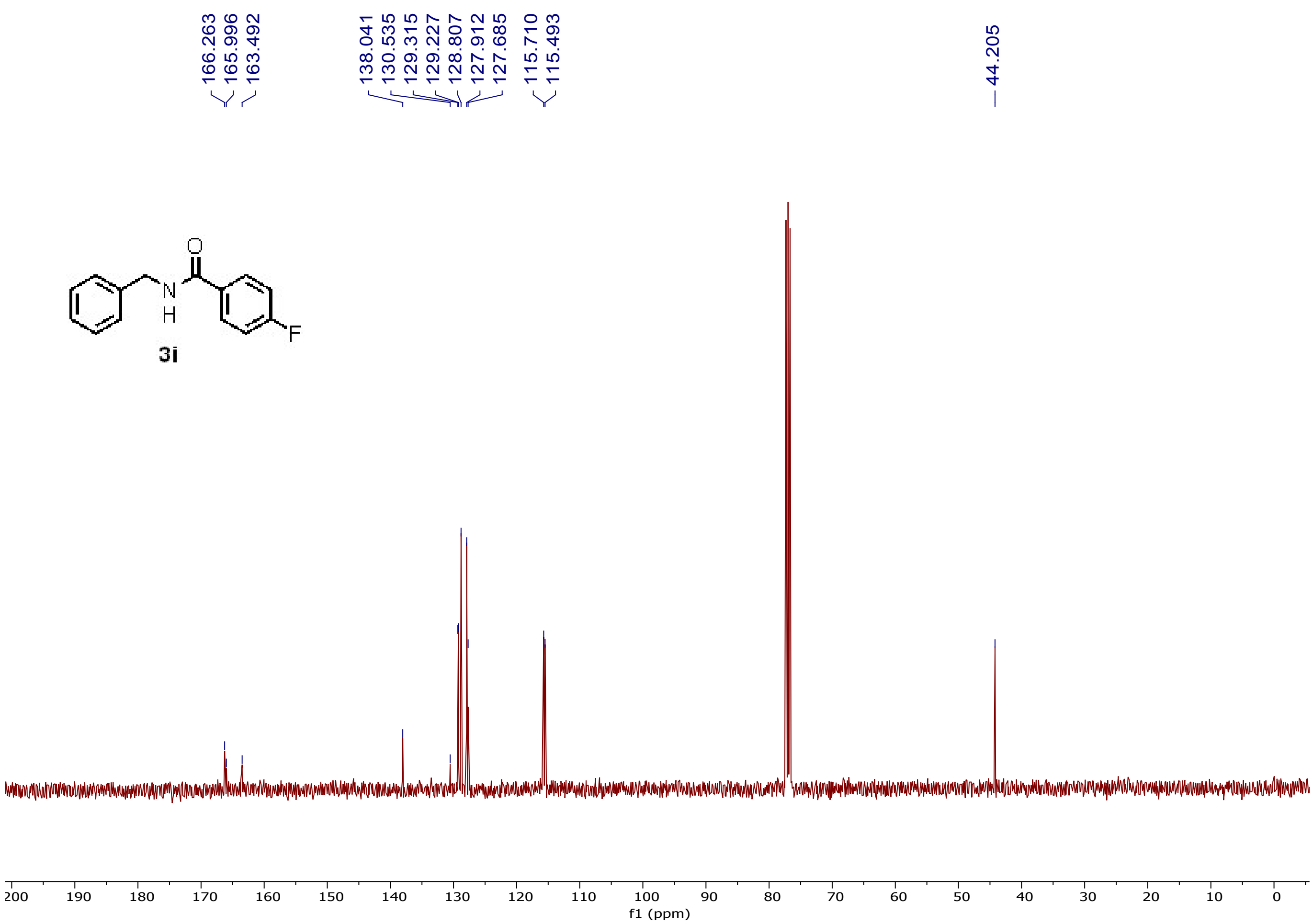

7.742
7.721
7.436
7.415
7.338
7.327
7.305
7.294
7.285
7.274
7.263
7.252
6.498
4.633
4.619
2.030
1.321
O
N
H
tBu
3g
2.00
2.04
5.03
0.98
2.09
9.26
f1 (ppm)

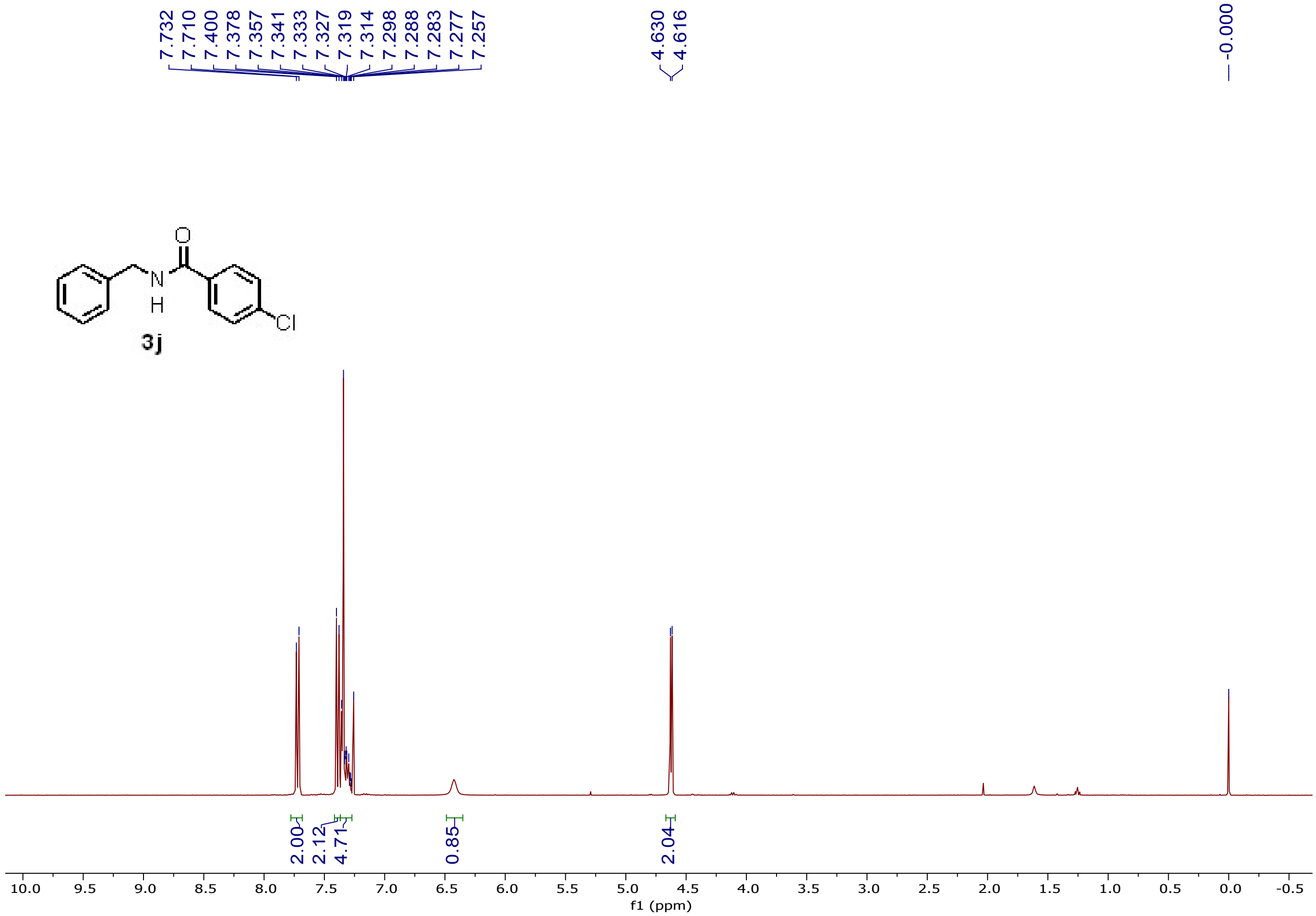

167.246
155.006
138.358
131.474
128.701
127.815
127.492
126.785
125.467
77.317
77.000
76.682
43.987
34.874
31.120
O
N
H
tBu
3g
f1 (ppm)

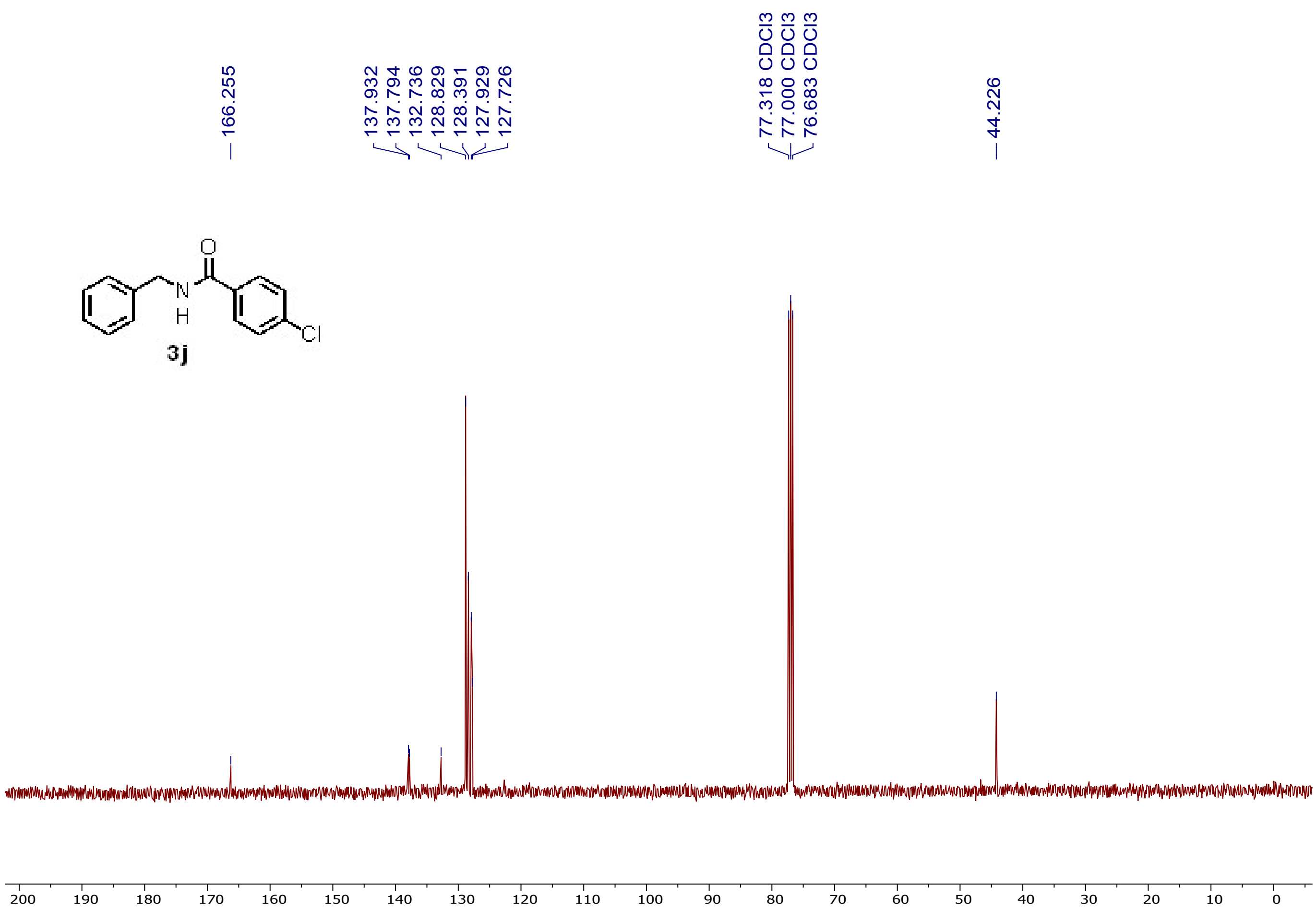

7.767
7.745
7.350
7.340
7.316
7.305
7.295
7.284
7.274
7.257
6.918
6.896
6.375
4.632
4.618
3.836
1.663
0.000
OMe
N
H
O
3h
2.00
5.14
1.99
0.96
2.04
3.07
f1 (ppm)

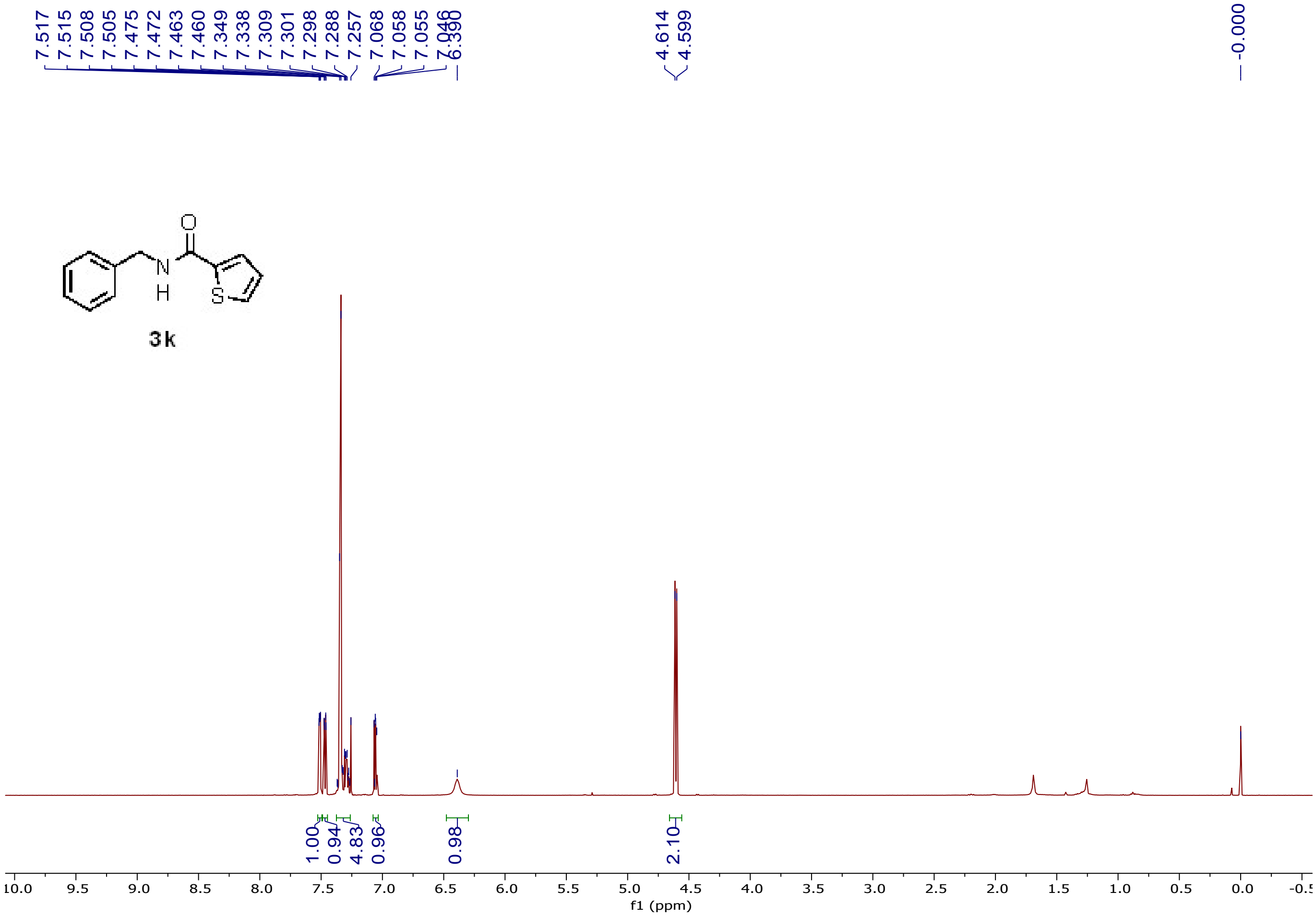

66.834
62.214
138.399
128.739
127.885
127.529
126.651
113.751
77.317 CDCl3
77.000 CDCl3
76.682 CDCl3
55.378
44.059
OMe
N
H
O
3h
f1 (ppm)

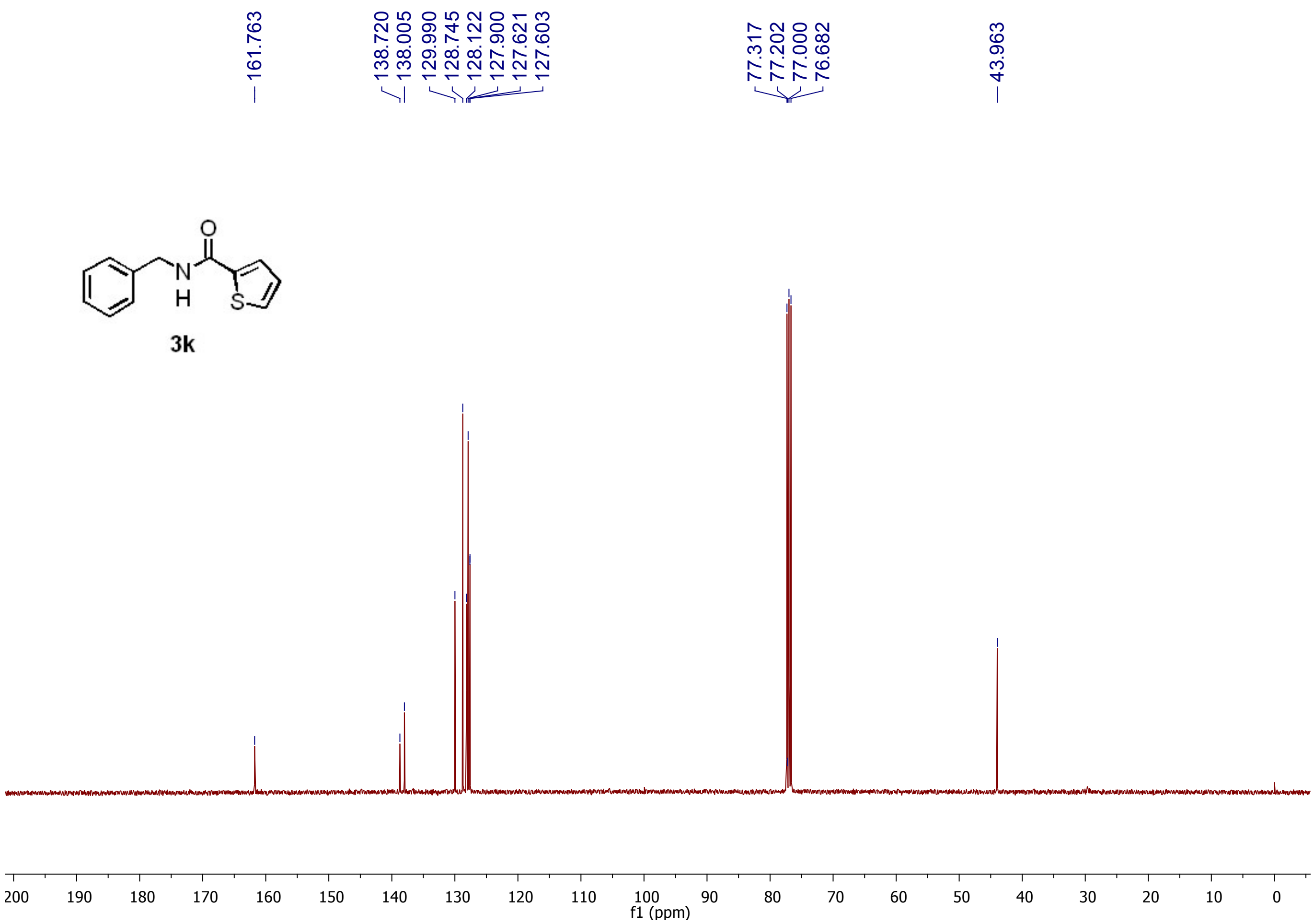

7.819
7.812
7.807
7.799
7.790
7.782
7.777
7.770
7.375
7.370
7.356
7.353
7.350
7.342
7.333
7.329
7.317
7.310
7.307
7.295
7.284
7.274
7.257
7.119
7.111
7.106
7.090
7.073
7.068
7.061
6.414
4.631
4.617
O
N
H
F
3i
2.00
5.15
2.02
0.98
2.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

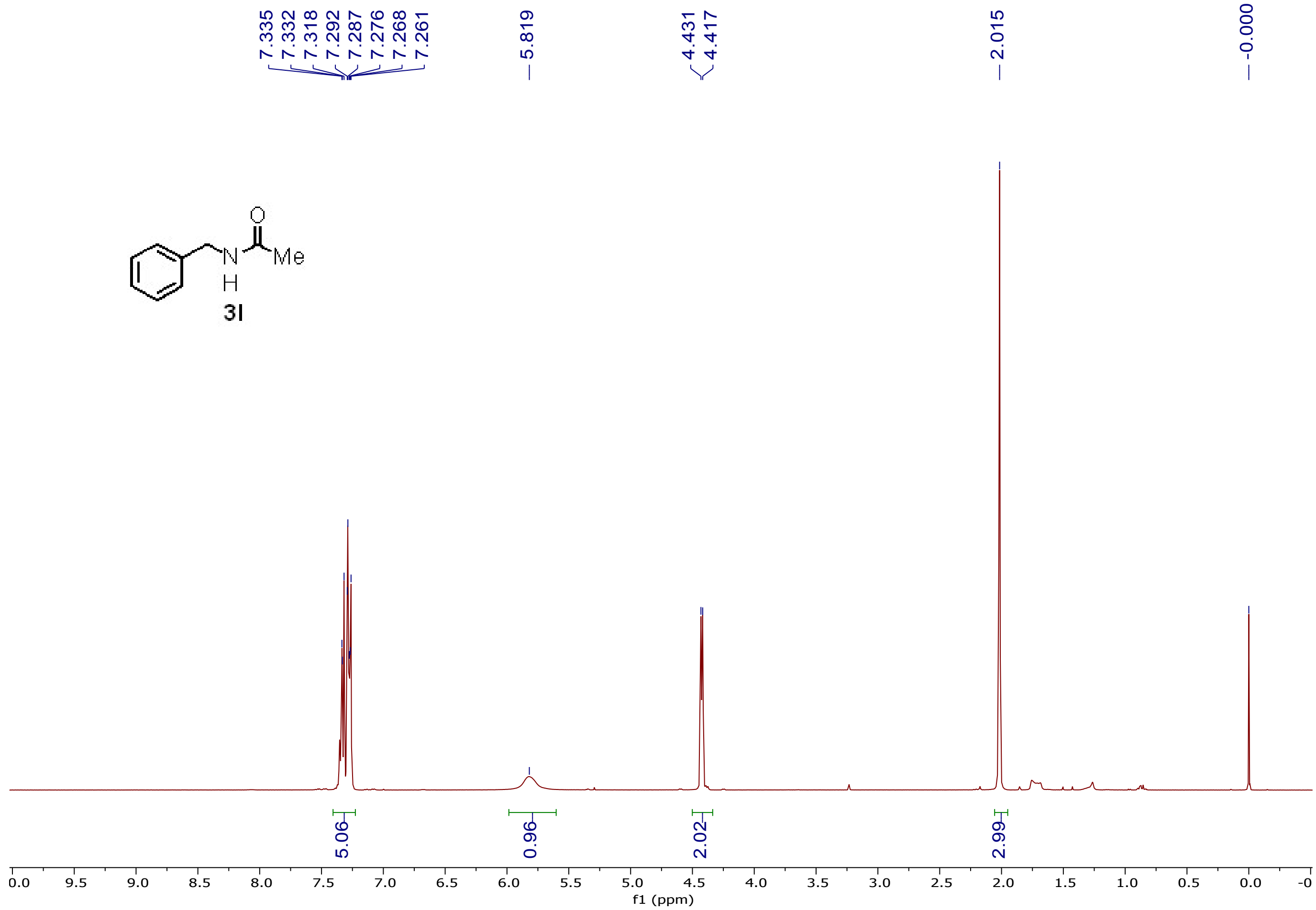

166.263
165.996
163.492
138.041
130.535
129.315
129.227
128.807
127.912
127.685
115.710
115.493
44.205
O
N
H
F
3i
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

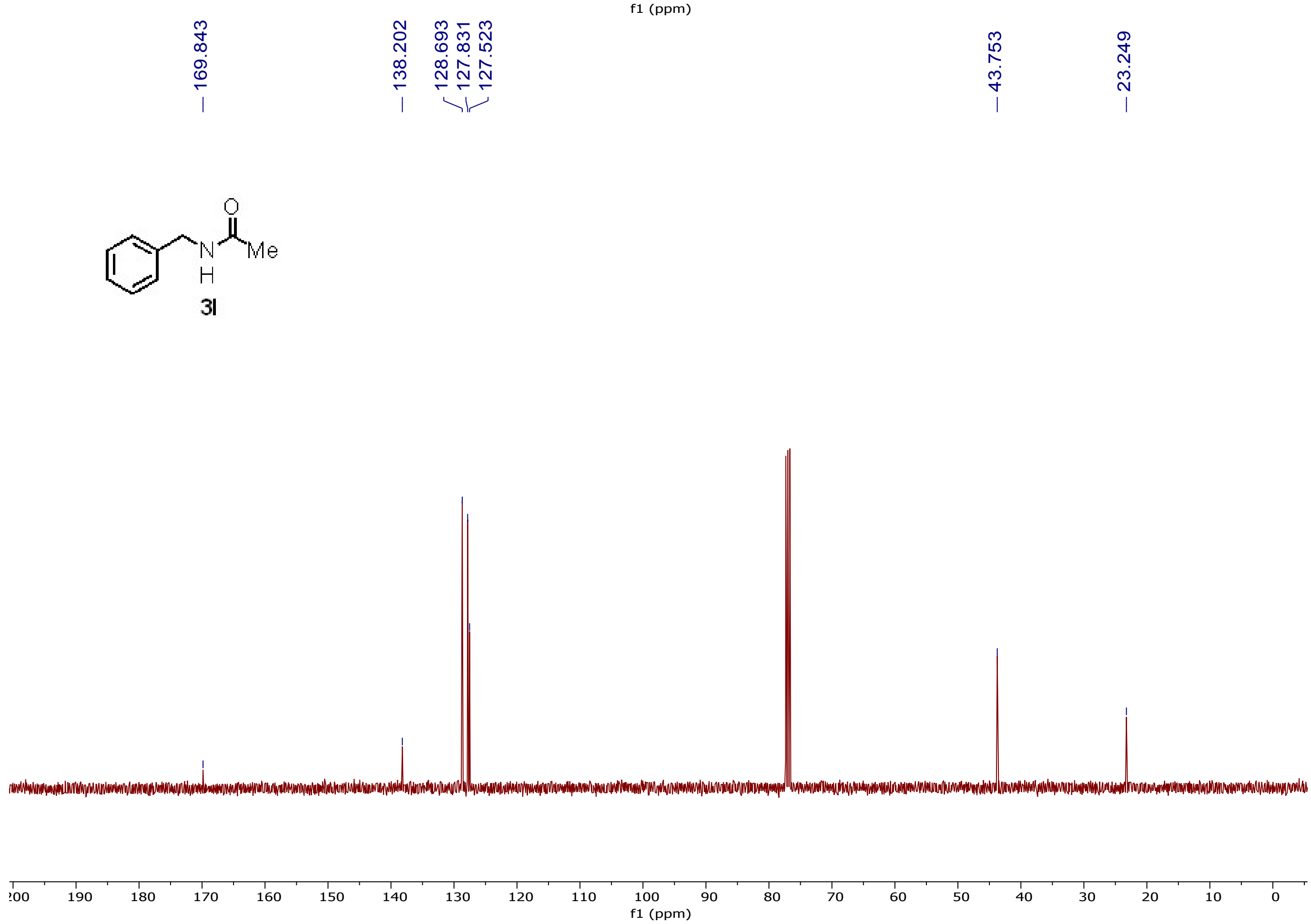

7.732
7.710
7.400
7.378
7.357
7.341
7.333
7.327
7.319
7.314
7.298
7.288
7.283
7.277
7.257
4.630
4.616
-0.000
O
N
H
Cl
3j
2.00
2.12
4.71
0.85
2.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

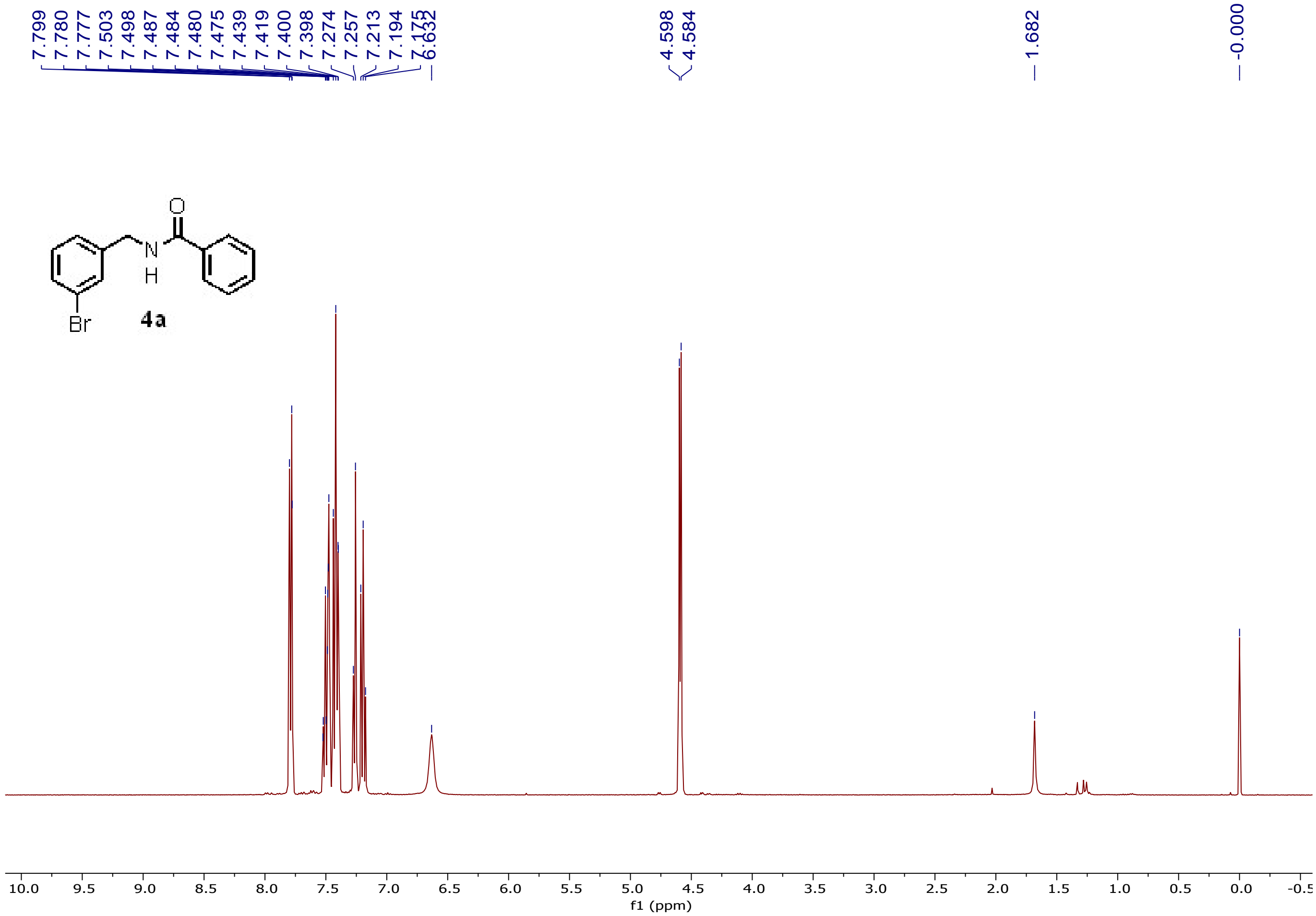

166.255
137.932
137.794
132.736
128.829
128.391
127.929
127.726
77.318 CDCl3
77.000 CDCl3
76.683 CDCl3
44.226
O
N
H
Cl
3j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

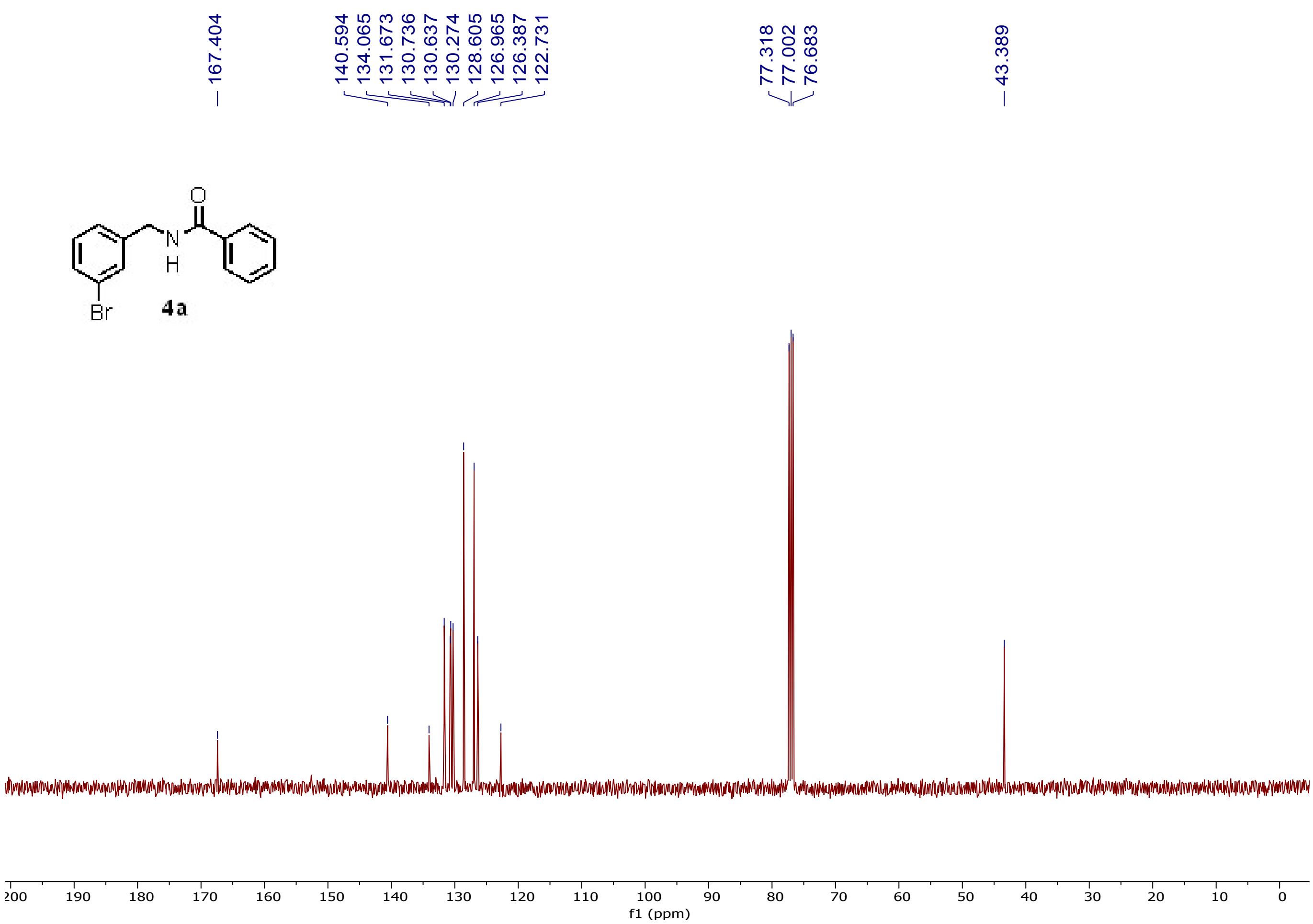

7.517
7.515
7.508
7.505
7.475
7.472
7.463
7.460
7.349
7.338
7.309
7.301
7.298
7.288
7.257
7.068
7.058
7.055
7.046
6.390
4.614
4.599
-0.000
O
N
H
S
3k
1.00
0.94
4.83
0.96
0.98
2.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

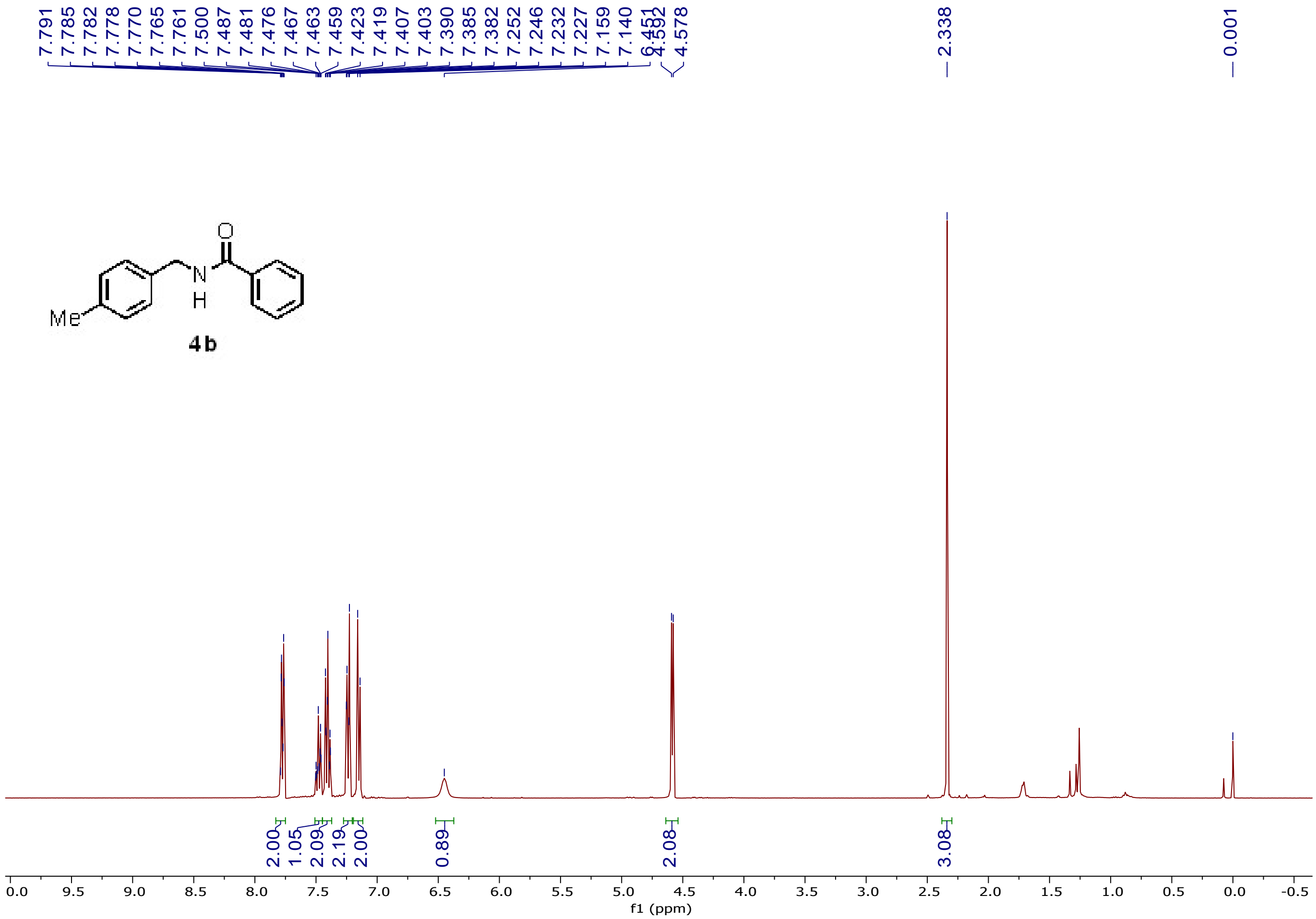

161.763
138.720
138.005
129.990
128.745
128.122
127.900
127.621
127.603
77.317
77.202
77.000
76.682
43.963
O
N
H
S
3k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

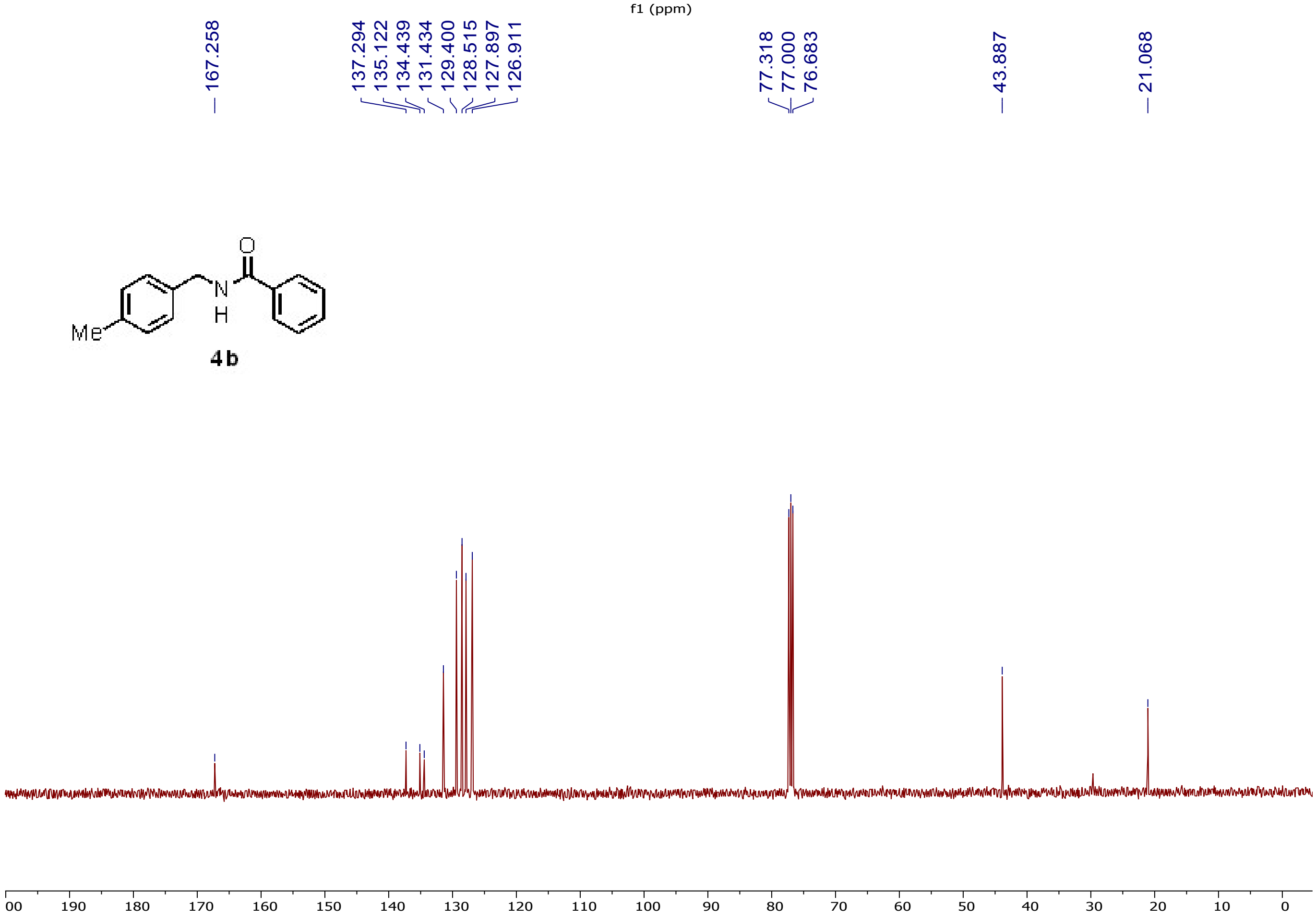

7.335
7.332
7.318
7.292
7.287
7.276
7.268
7.261
5.819
4.431
4.417
2.015
-0.000
O
N
H
Me
3l
5.06
0.96
2.02
2.99
f1 (ppm)

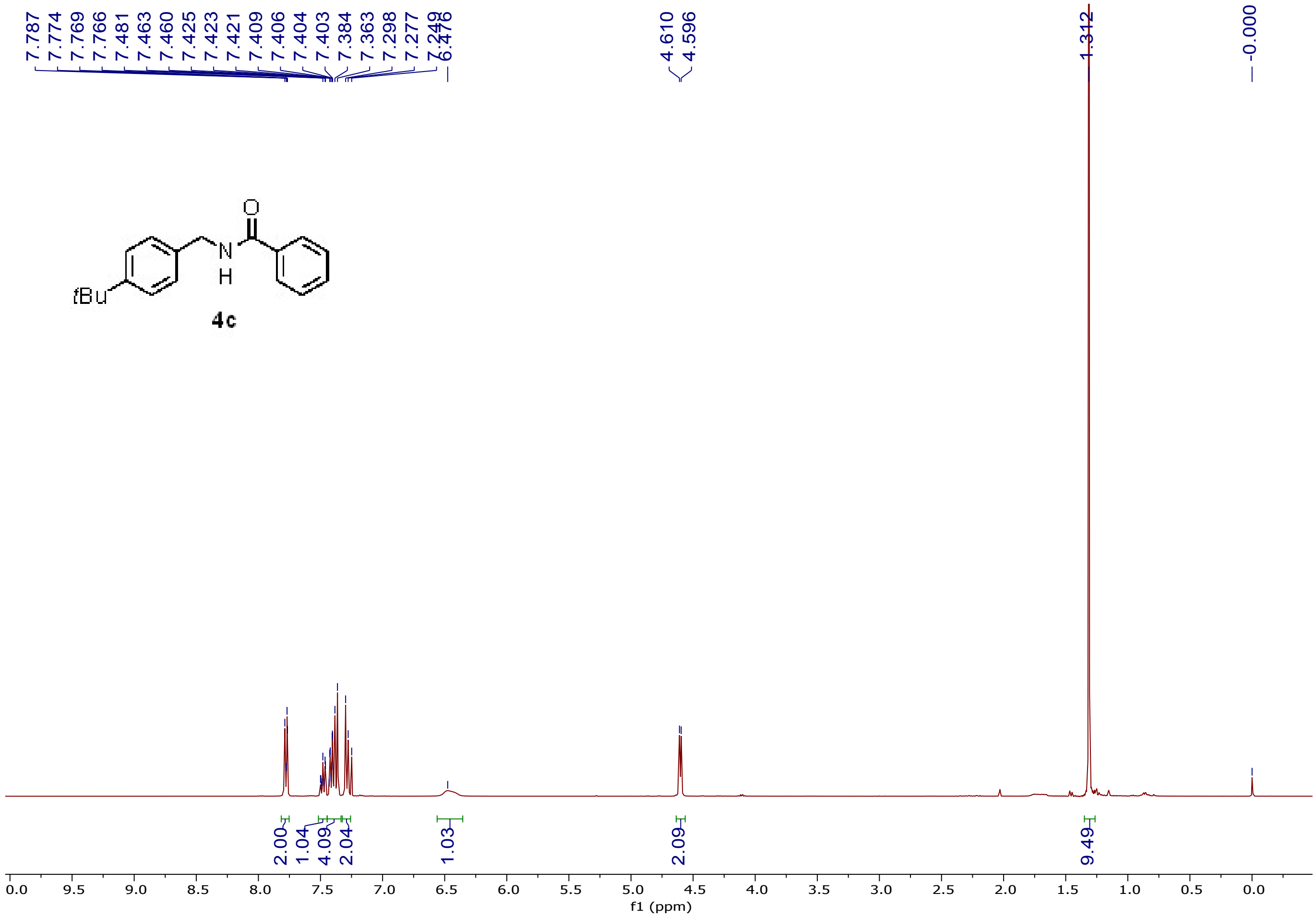

169.843
138.202
128.693
127.831
127.523
43.753
23.249
O
N
H
Me
3l
f1 (ppm)

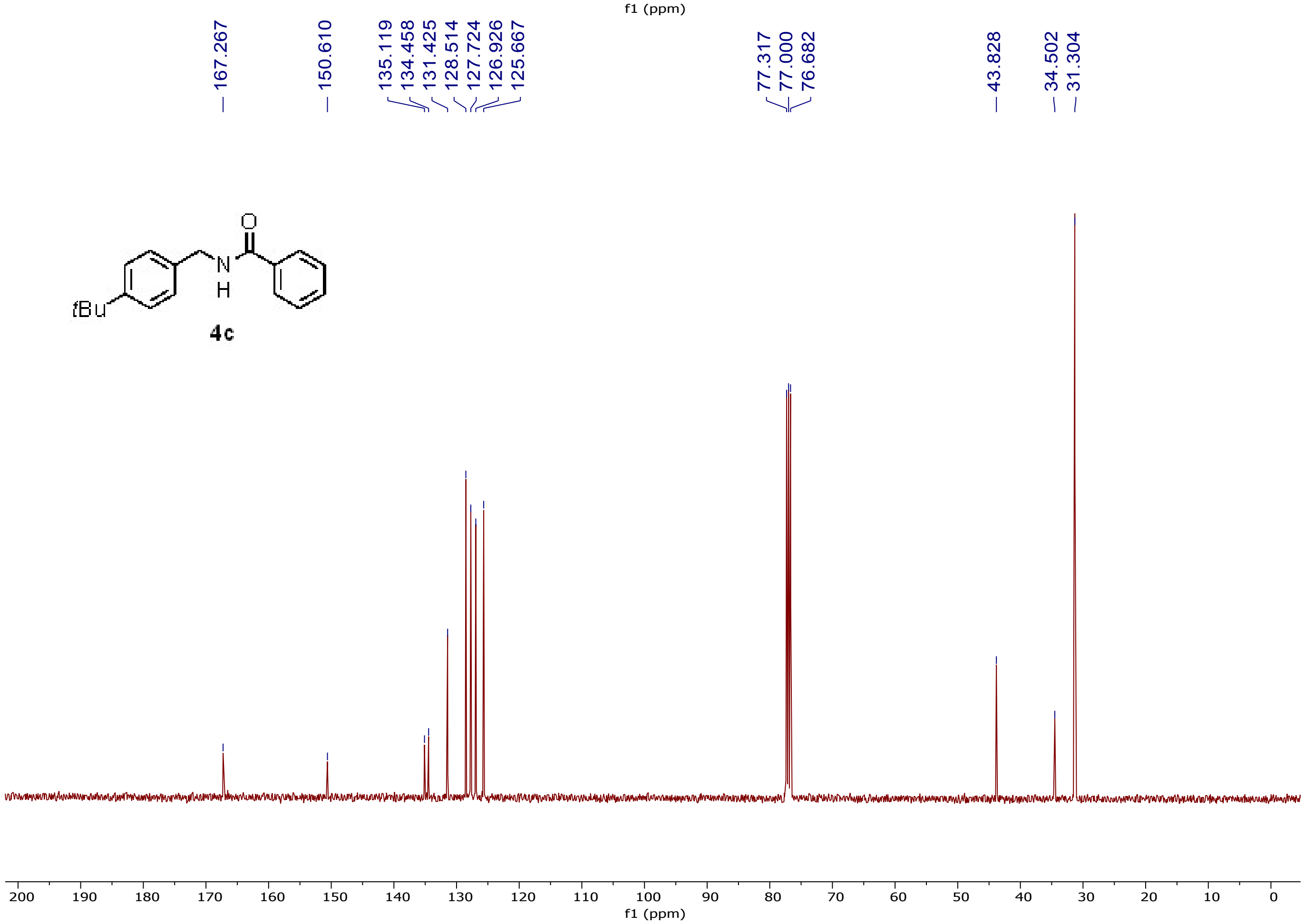

7.799 7.780 7.777 7.503 7.498 7.487 7.484 7.480 7.475 7.439 7.419 7.400 7.398 7.274 7.257 7.213 7.194 7.175 6.632
4.598 4.584
1.682
-0.000
Br
4a
f1 (ppm)

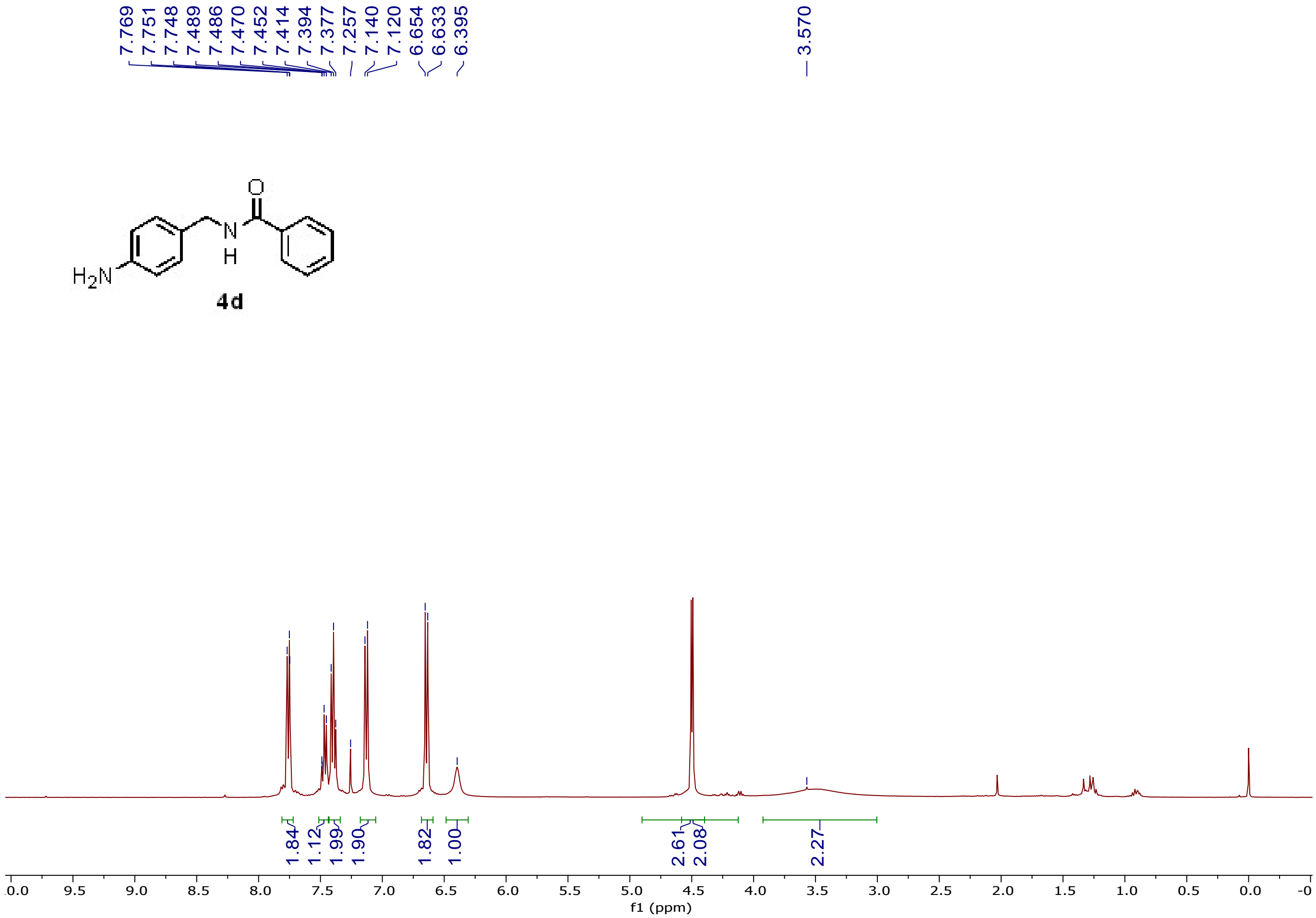

167.404
140.594 134.065 131.673 130.736 130.637 130.274 128.605 126.965 126.387 122.731
77.318 77.002 76.683
43.389
Br
4a
f1 (ppm)

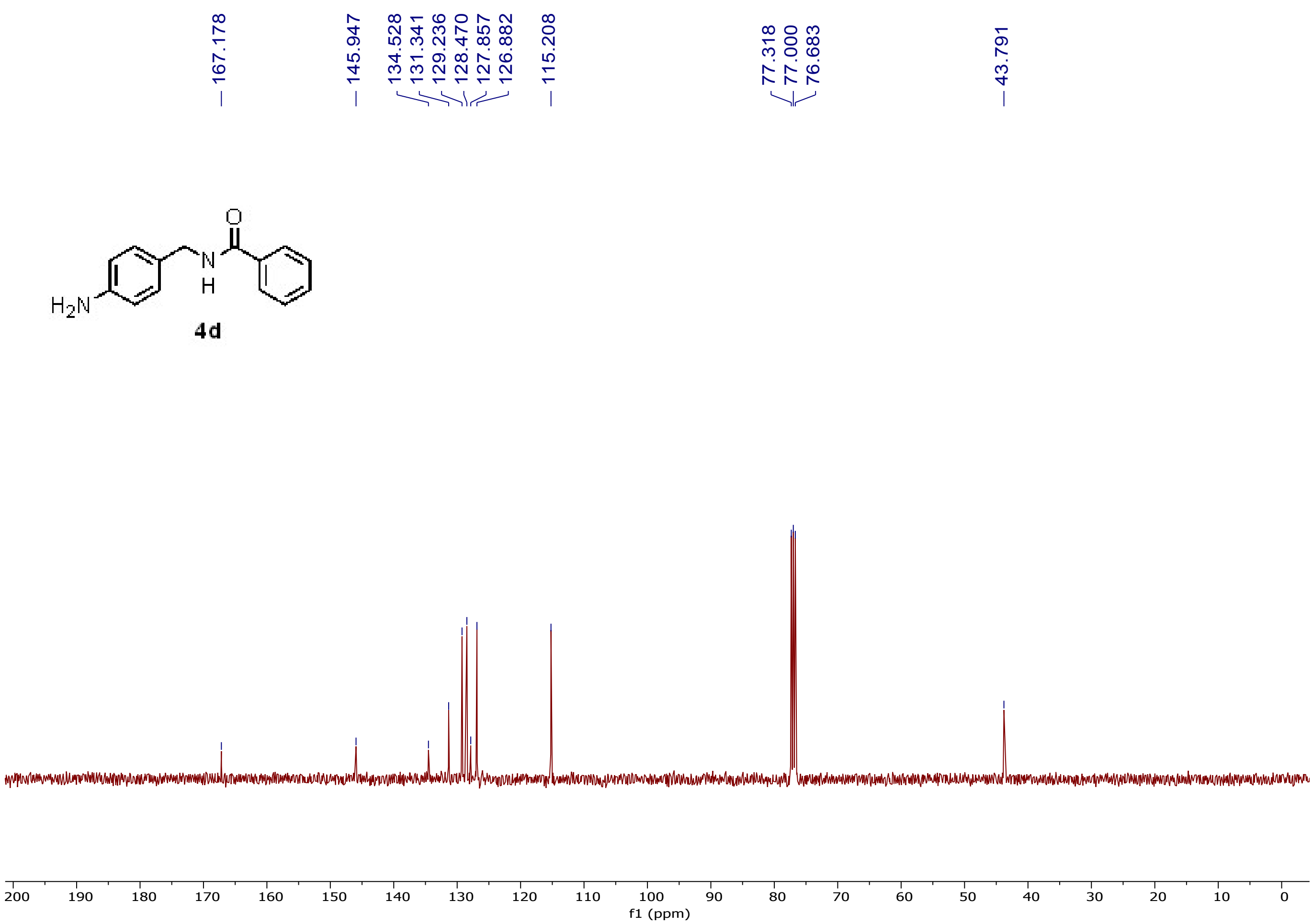

7.791
7.785
7.782
7.778
7.770
7.765
7.761
7.500
7.487
7.481
7.476
7.467
7.463
7.459
7.423
7.419
7.407
7.403
7.390
7.385
7.382
7.252
7.246
7.232
7.227
7.159
7.140
6.451
4.592
4.578
2.338
0.001
O
N
H
Me
4b
2.00
1.05
2.09
2.19
2.00
0.89
2.08
3.08
f1 (ppm)

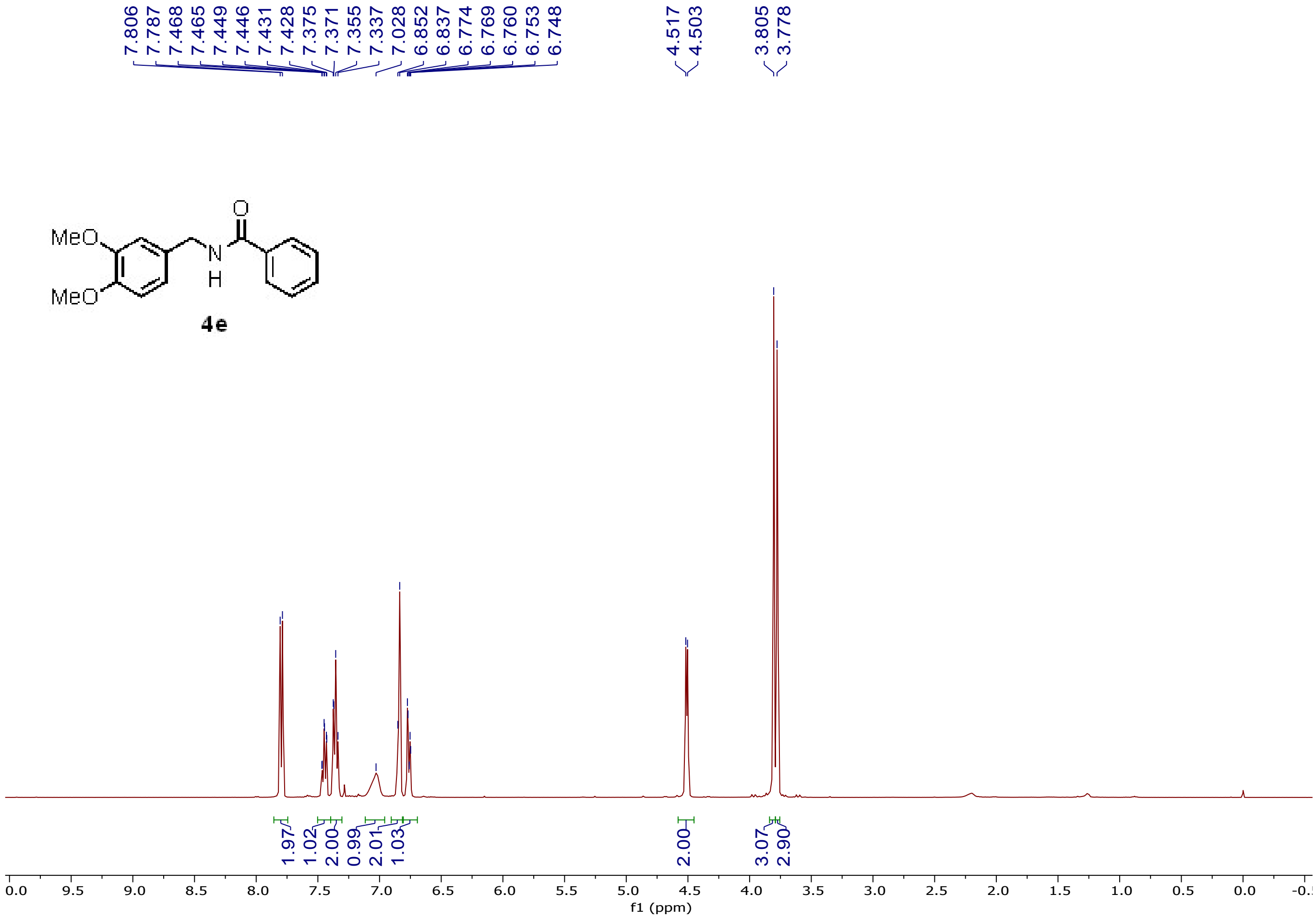

167.258
137.294
135.122
134.439
131.434
129.400
128.515
127.897
126.911
77.318
77.000
76.683
43.887
21.068
O
N
H
Me
4b
f1 (ppm)

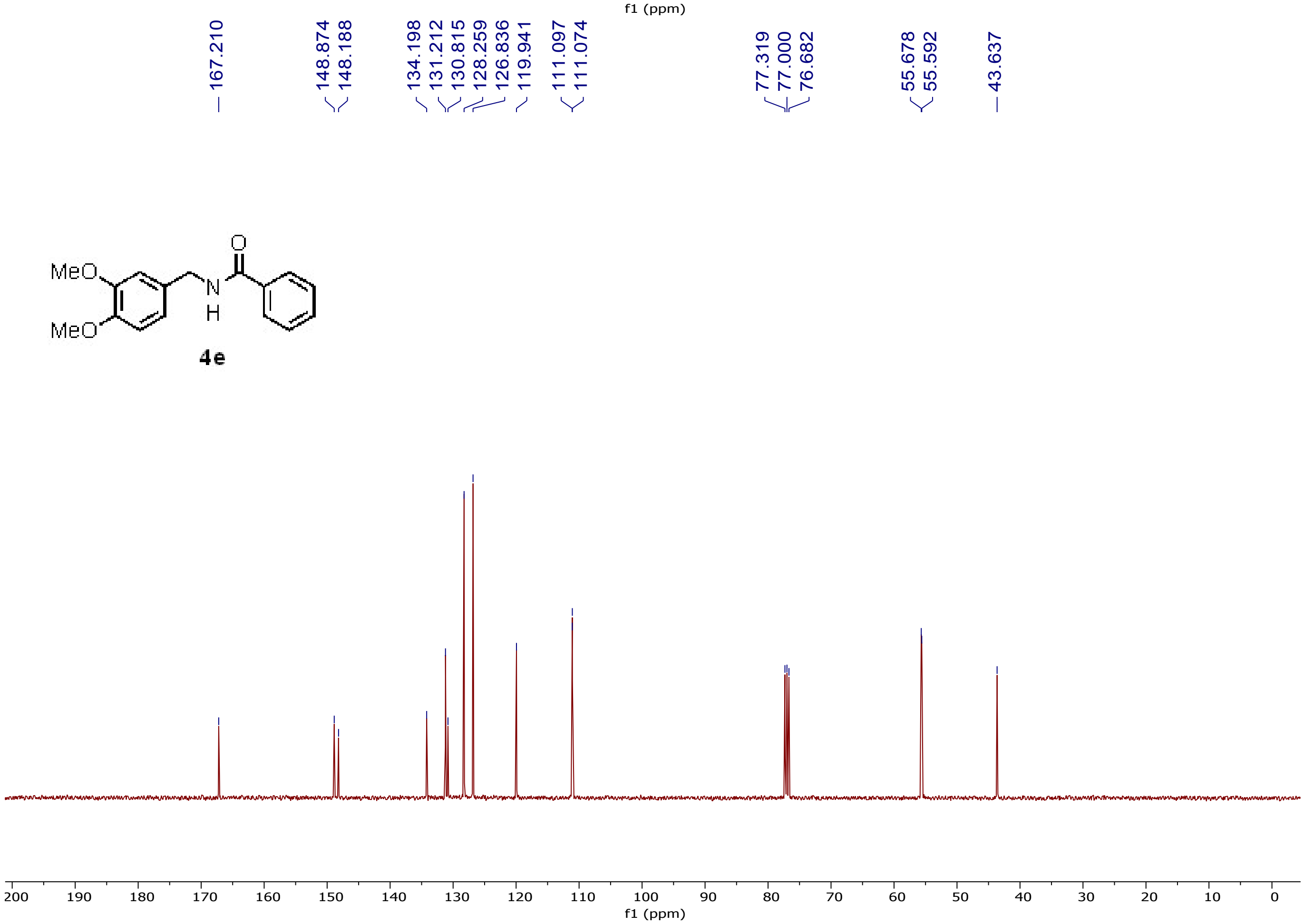

7.787
7.774
7.769
7.766
7.481
7.463
7.460
7.425
7.423
7.421
7.409
7.406
7.404
7.403
7.384
7.363
7.298
7.277
7.249
6.476
4.610
4.596
1.312
-0.000
O
N
H
tBu
4c
2.00
1.04
4.09
2.04
1.03
2.09
9.49
f1 (ppm)

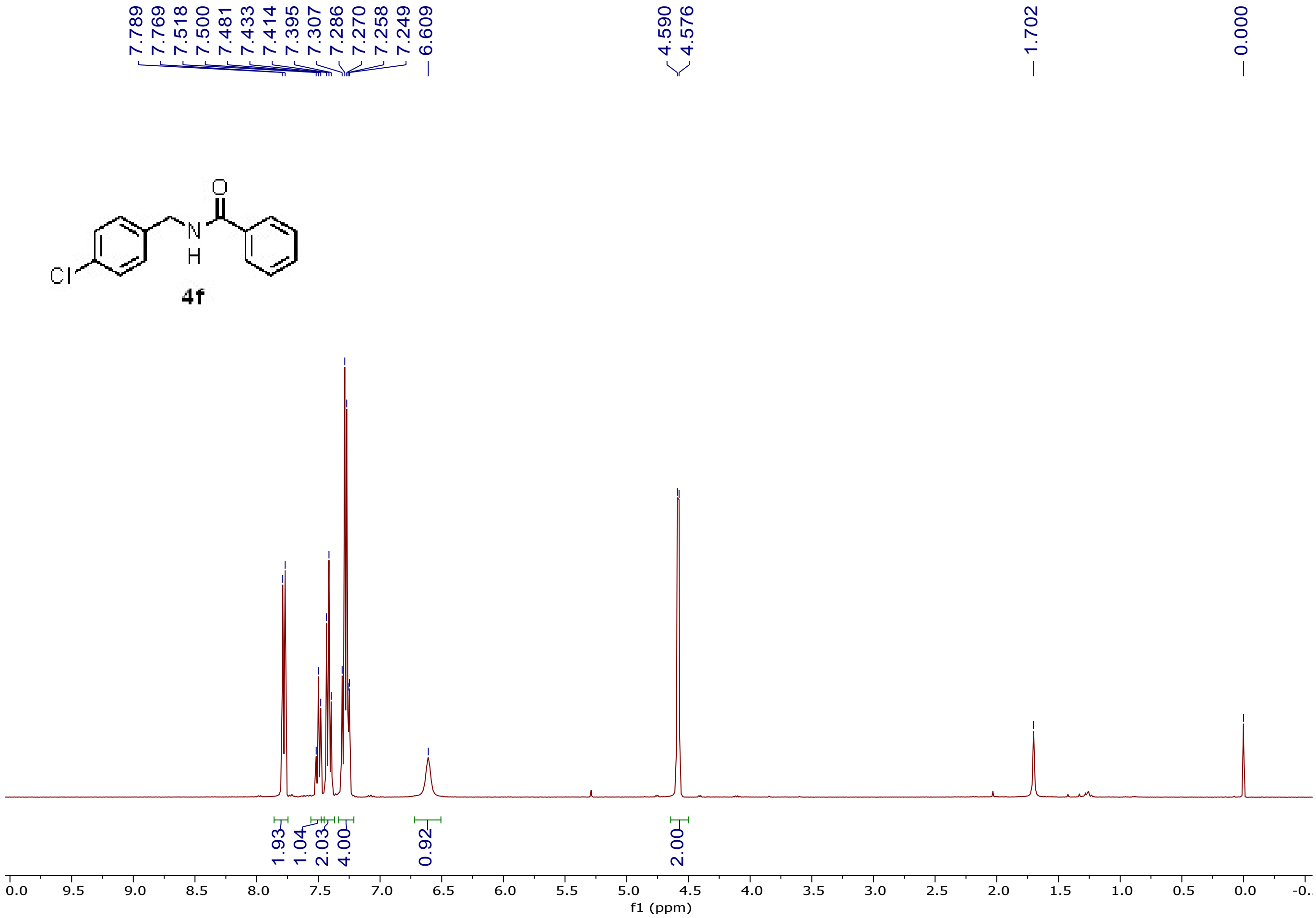

167.267
150.610
135.119
134.458
131.425
128.514
127.724
126.926
125.667
77.317
77.000
76.682
43.828
34.502
31.304
O
N
H
tBu
4c
f1 (ppm)

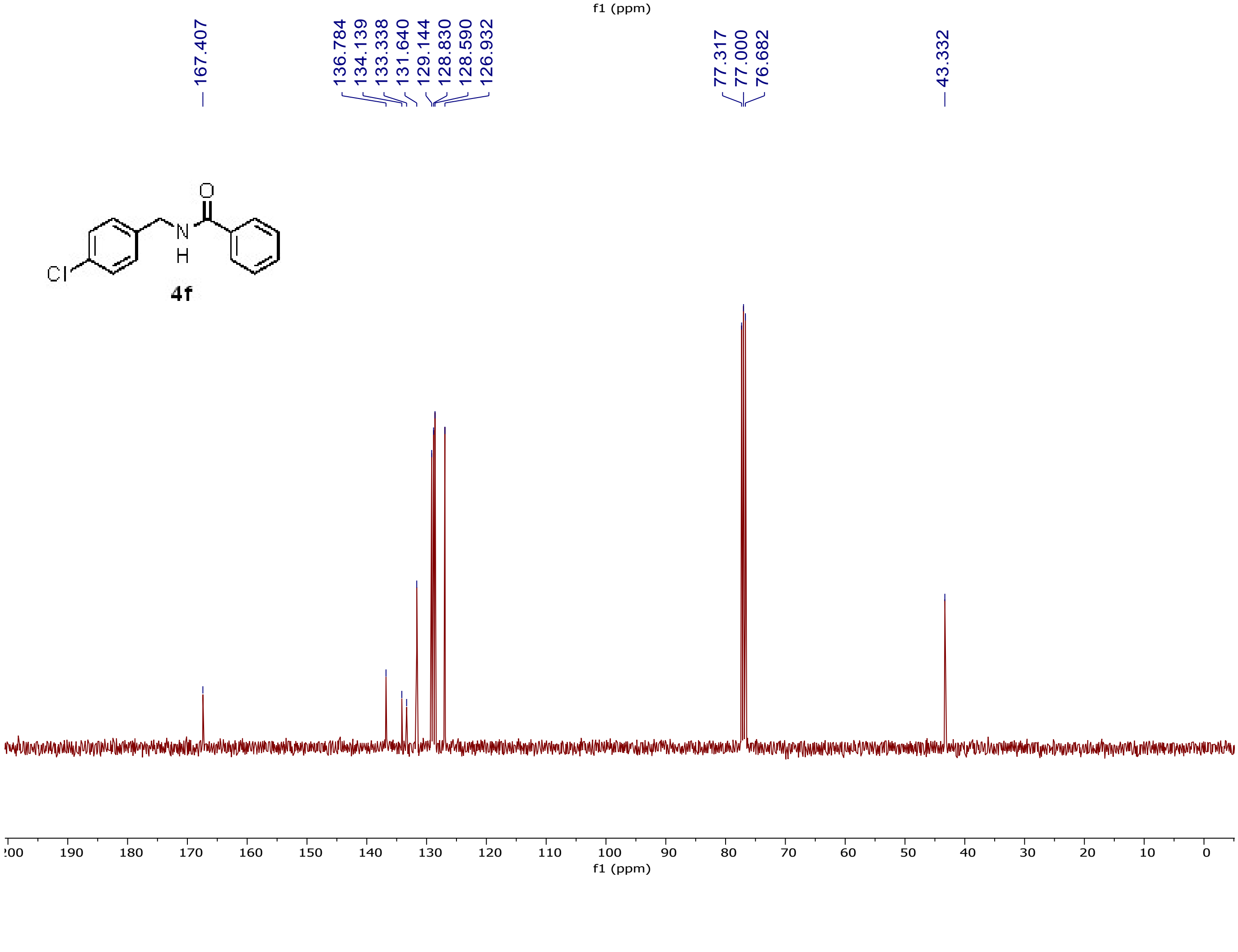

7.769
7.751
7.748
7.489
7.486
7.470
7.452
7.414
7.394
7.377
7.257
7.140
7.120
6.654
6.633
6.395
3.570
O
N
H
H_2N
4d
1.84
1.12
1.99
1.90
1.82
1.00
2.61
2.08
2.27
f1 (ppm)

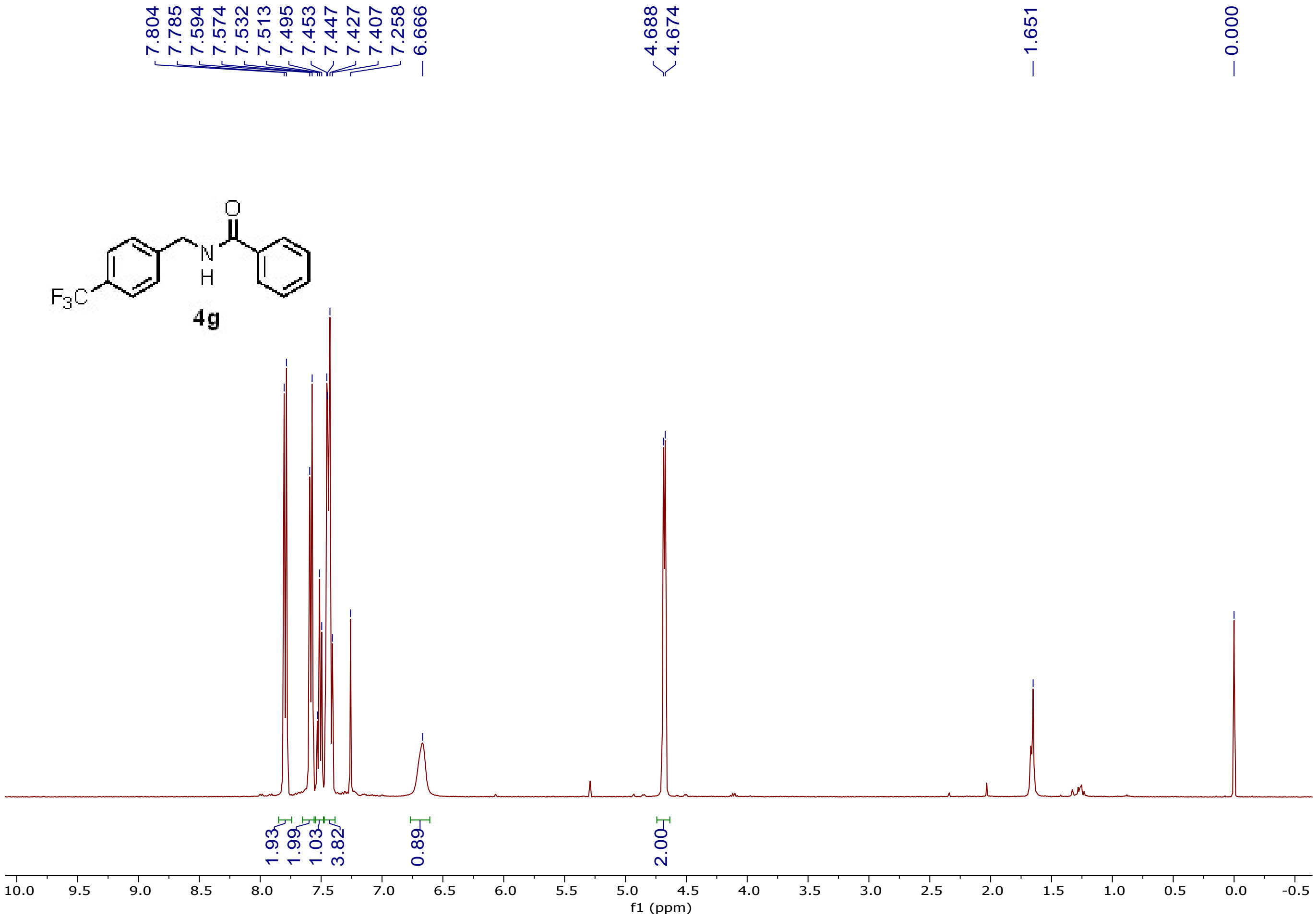

167.178
145.947
134.528
131.341
129.236
128.470
127.857
126.882
115.208
77.318
77.000
76.683
43.791
O
N
H
H_2N
4d
f1 (ppm)

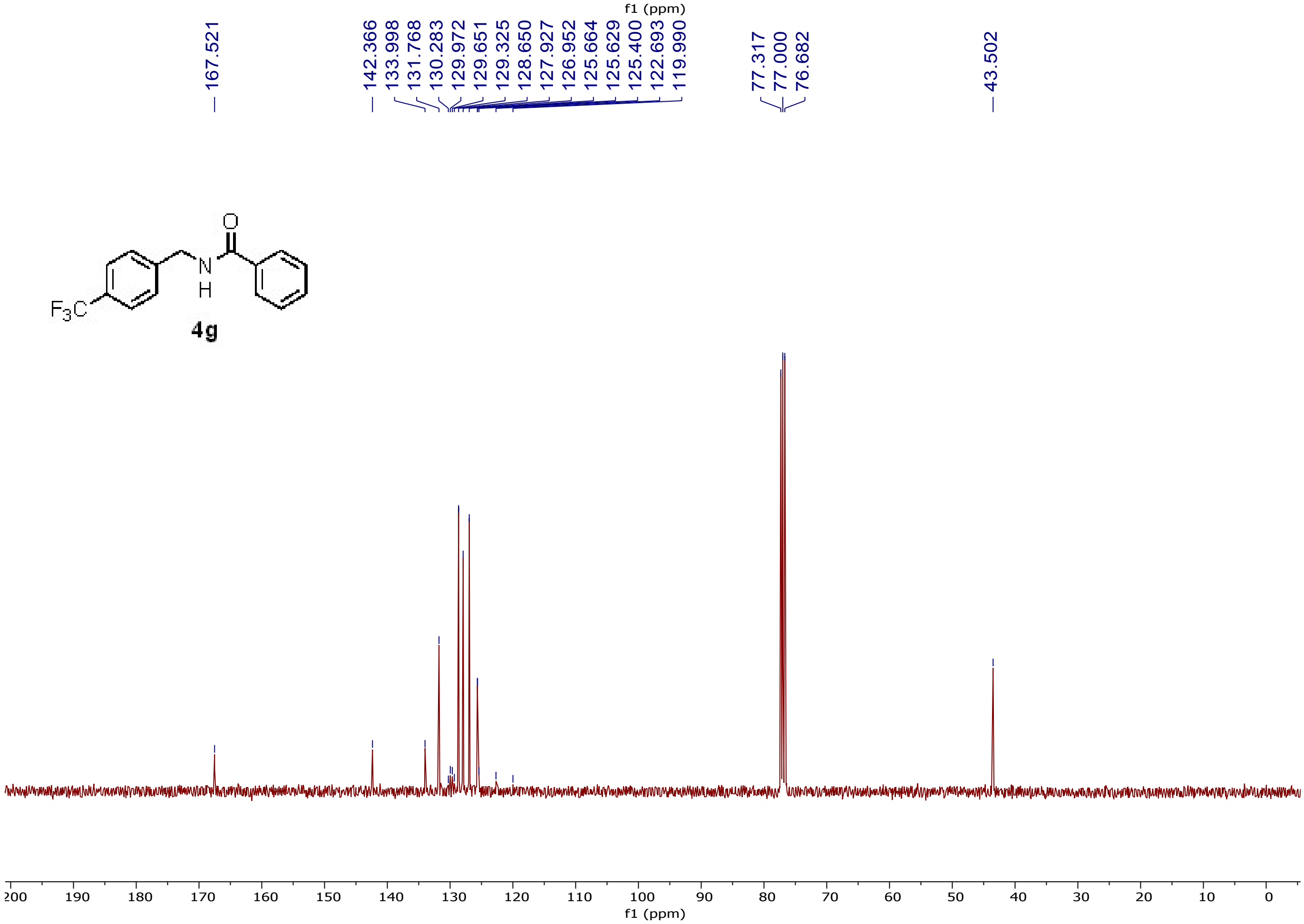

7.806
7.787
7.468
7.465
7.449
7.446
7.431
7.428
7.375
7.371
7.355
7.337
7.028
6.852
6.837
6.774
6.769
6.760
6.753
6.748
4.517
4.503
3.805
3.778
MeO
MeO
O
N
H
4e
1.97
1.02
2.00
0.99
2.01
1.03
2.00
3.07
2.90
f1 (ppm)

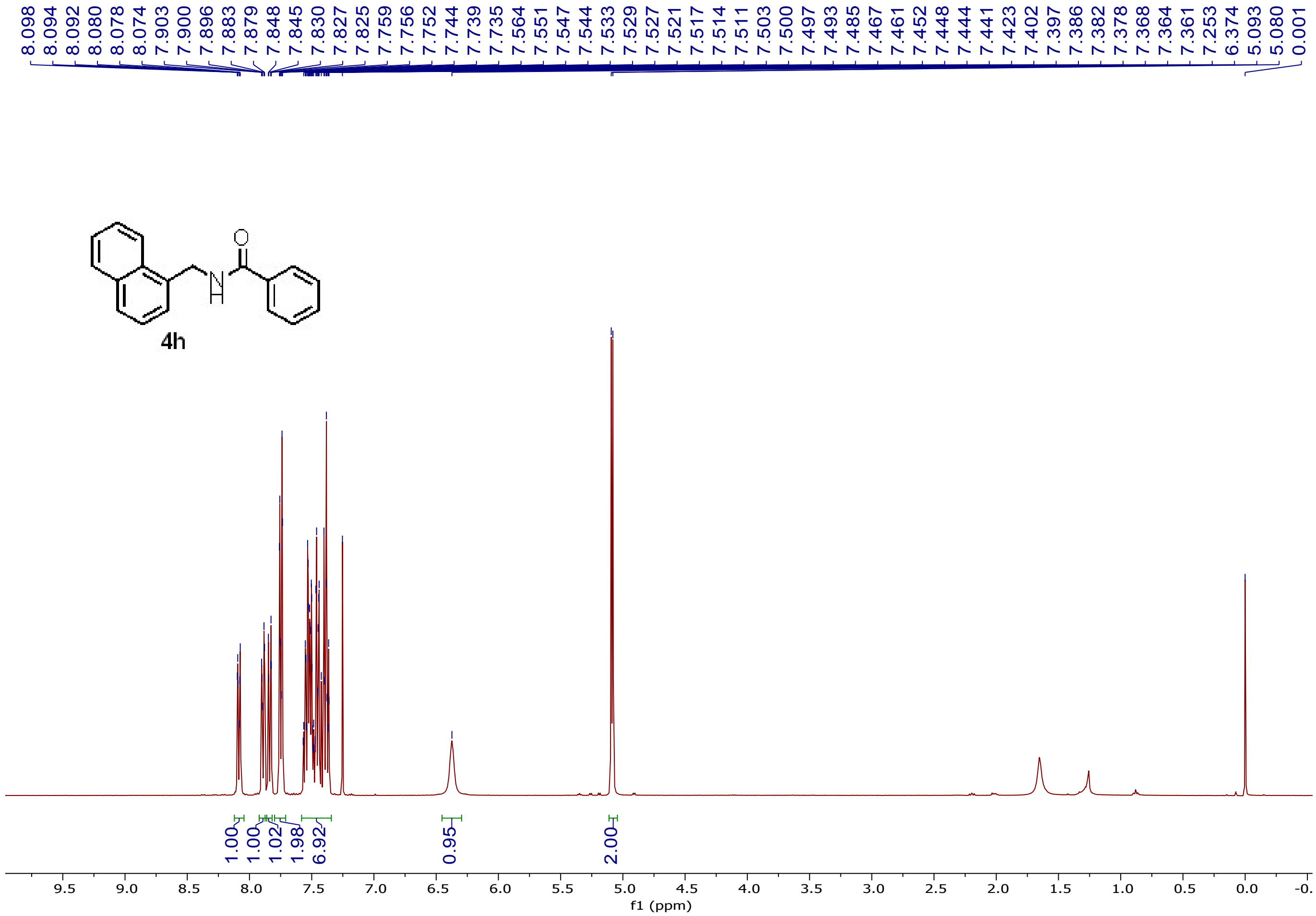

167.210
148.874
148.188
134.198
131.212
130.815
128.259
126.836
119.941
111.097
111.074
77.319
77.000
76.682
55.678
55.592
43.637
MeO
MeO
O
N
H
4e
f1 (ppm)

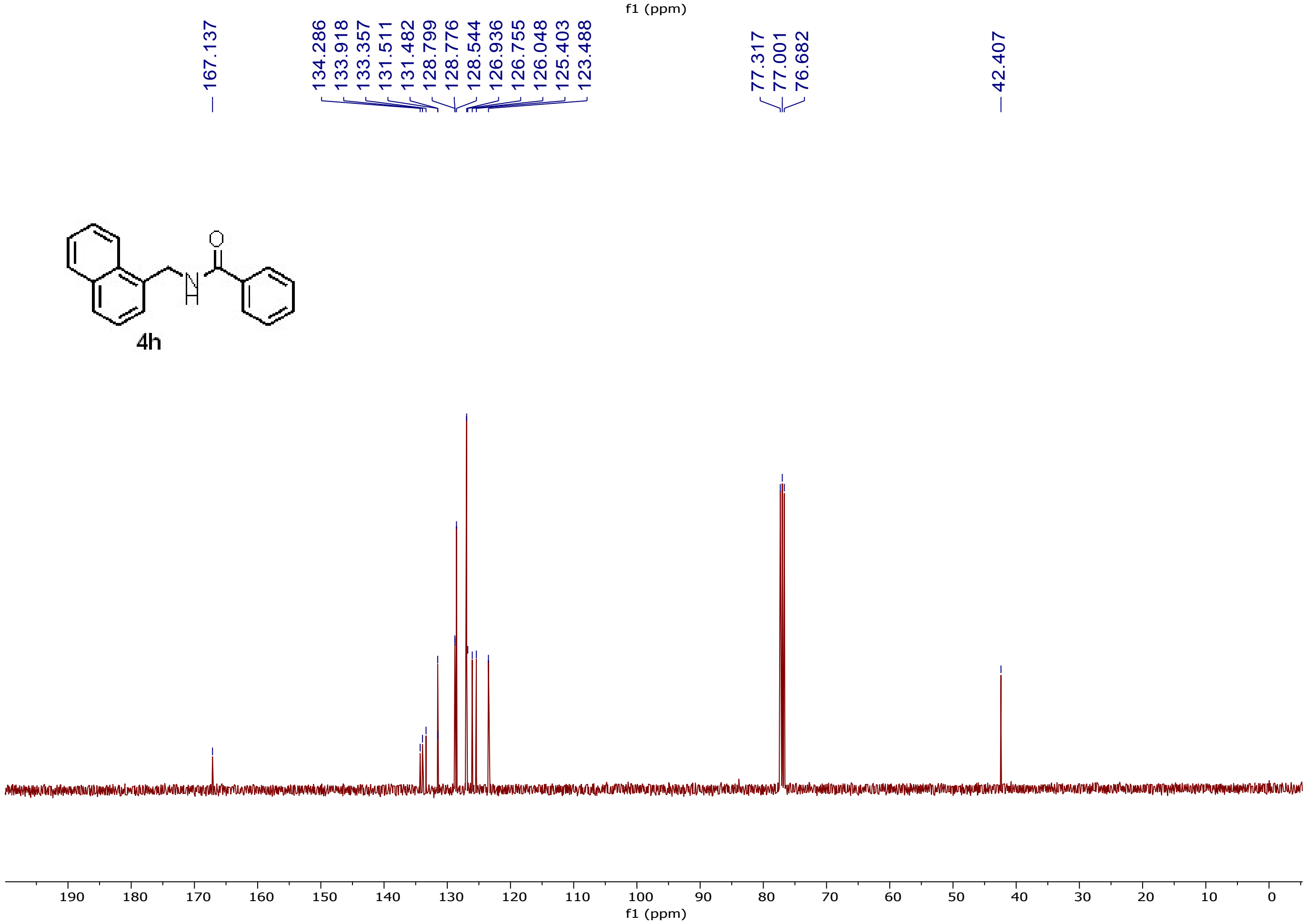

7.789
7.769
7.518
7.500
7.481
7.433
7.414
7.395
7.307
7.286
7.270
7.258
7.249
6.609
4.590
4.576
1.702
0.000
Cl
N
H
O
4f
1.93
1.04
2.03
4.00
0.92
2.00
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

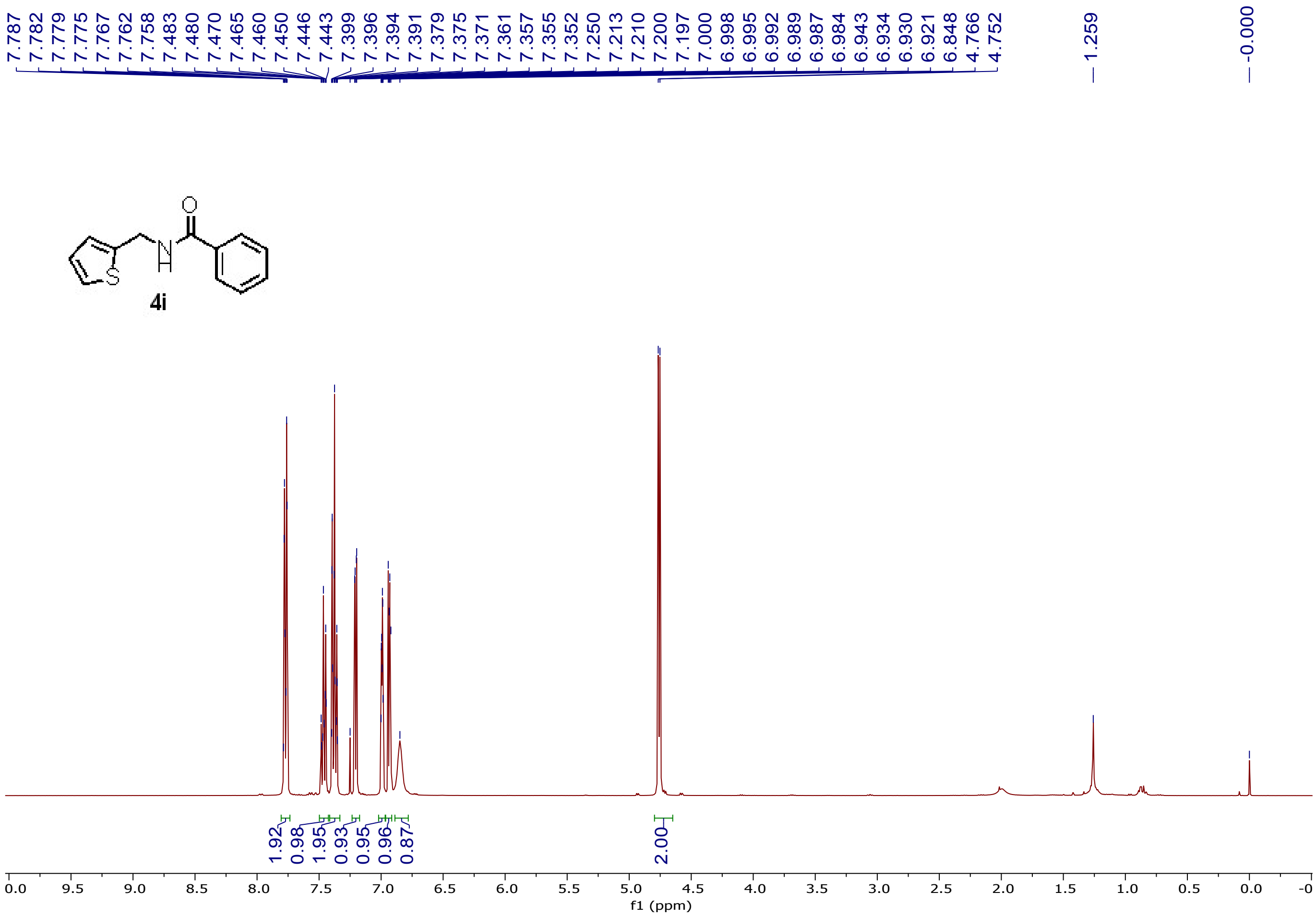

167.407
136.784
134.139
133.338
131.640
129.144
128.830
128.590
126.932
77.317
77.000
76.682
43.332
Cl
N
H
O
4f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

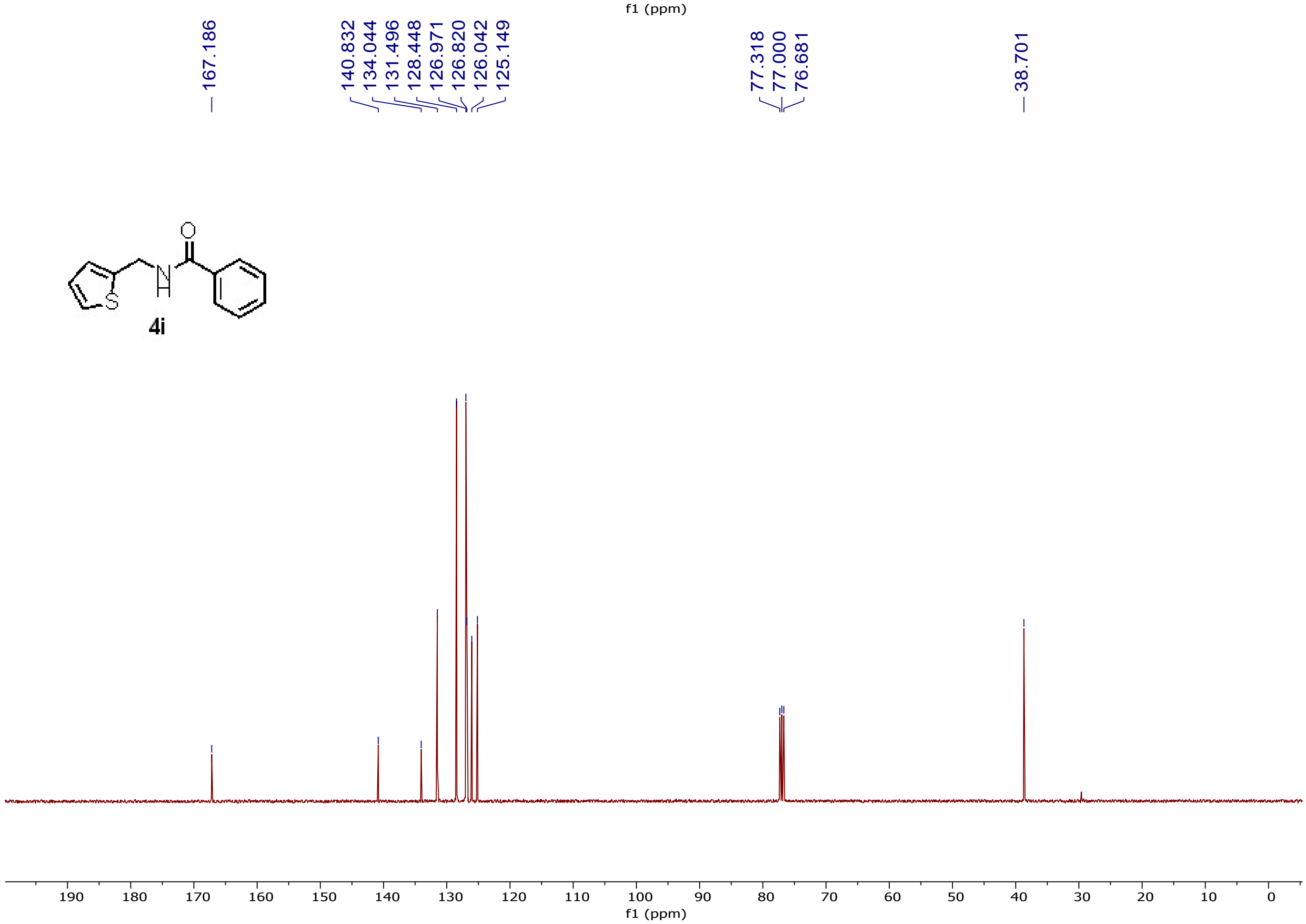

7.804
7.785
7.594
7.574
7.532
7.513
7.495
7.453
7.447
7.427
7.407
7.258
6.666
4.688
4.674
1.651
0.000
O
N
H
F3C
4g
1.93
1.99
1.03
3.82
0.89
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

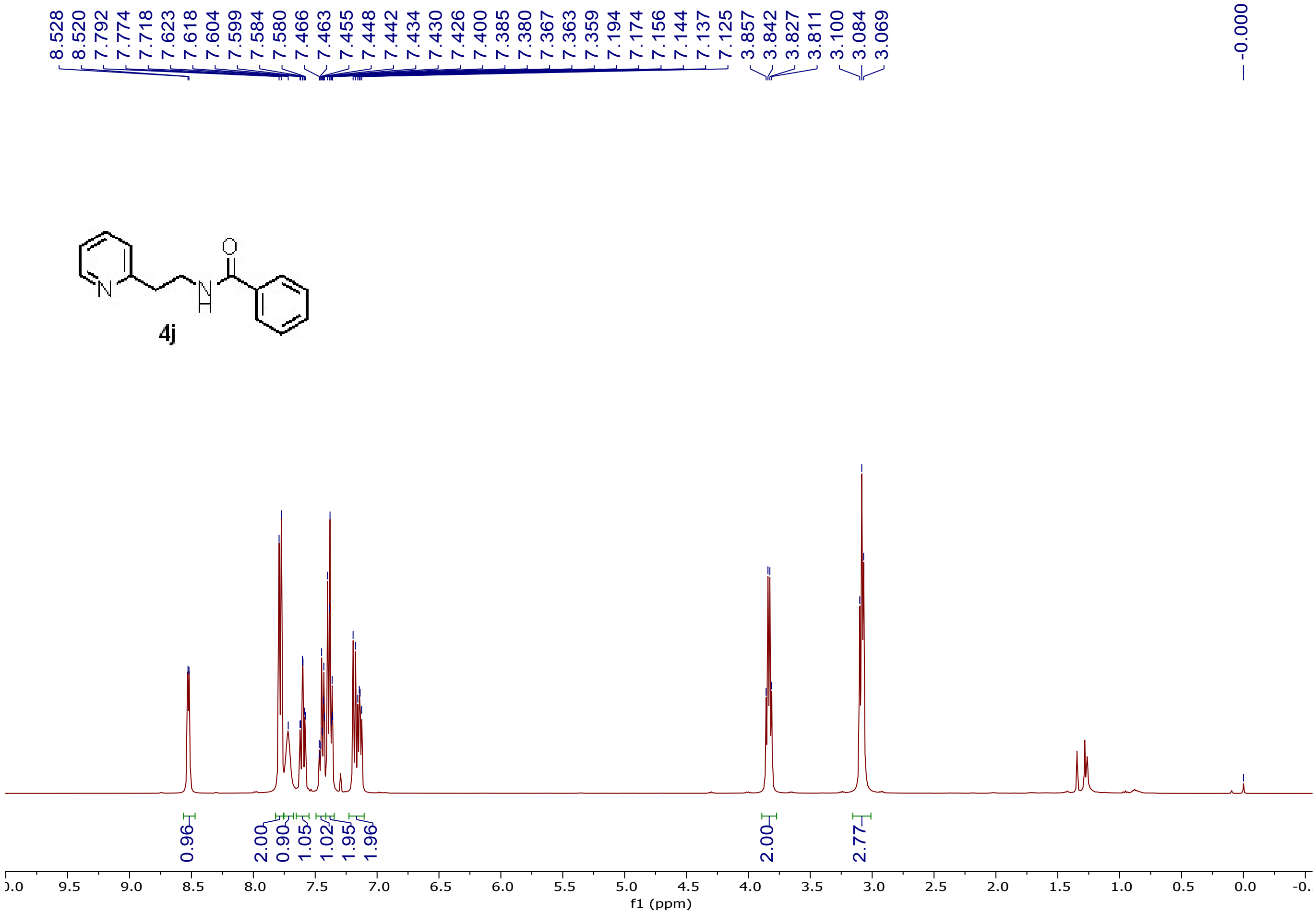

167.521
142.366
133.998
131.768
130.283
129.972
129.651
129.325
128.650
127.927
126.952
125.664
125.629
125.400
122.693
119.990
77.317
77.000
76.682
43.502
O
N
H
F3C
4g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

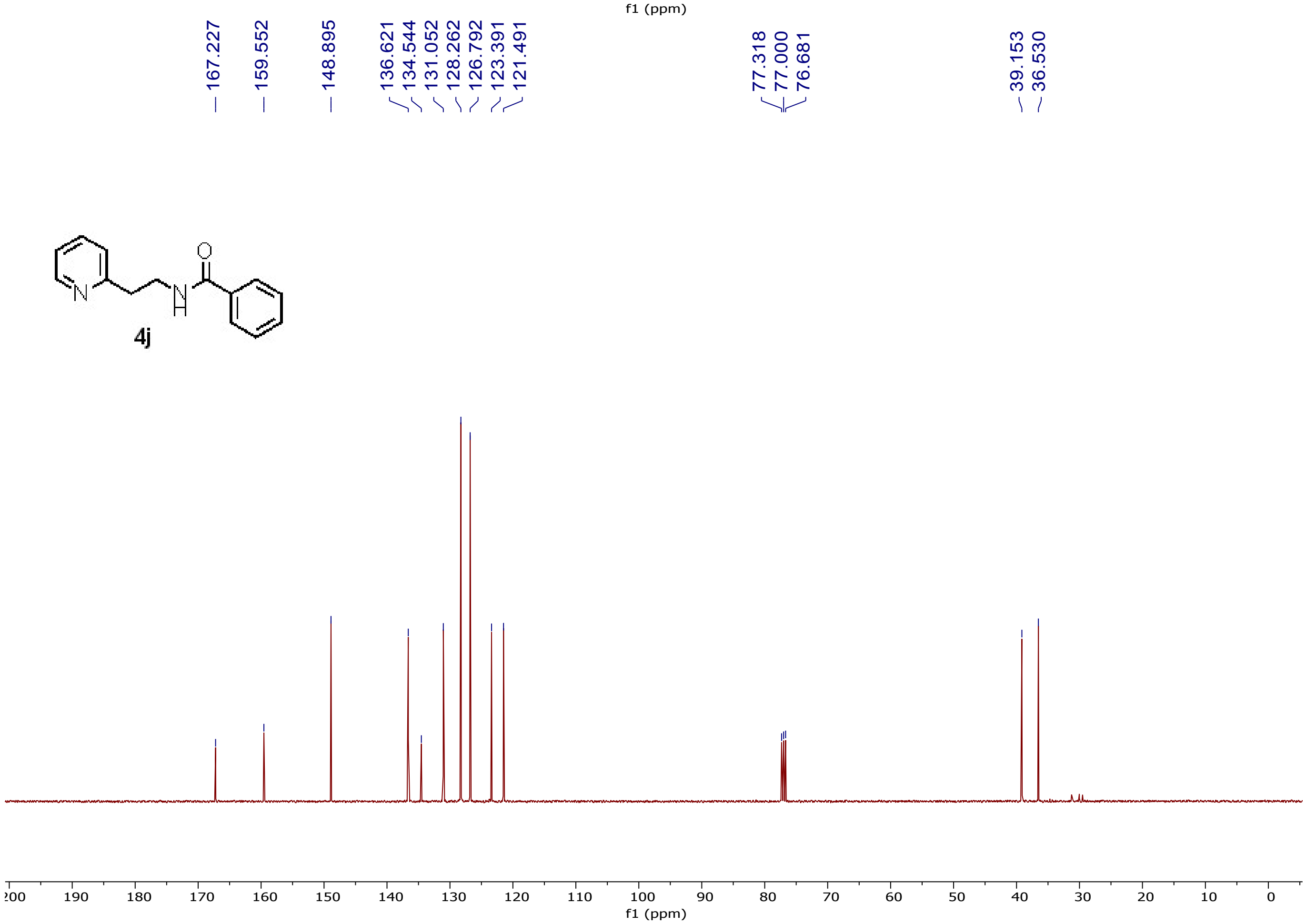

8.098 8.094 8.092 8.080 8.078 8.074 7.903 7.900 7.896 7.883 7.879 7.848 7.845 7.830 7.827 7.825 7.759 7.756 7.752 7.744 7.739 7.735 7.564 7.551 7.547 7.544 7.533 7.529 7.527 7.521 7.517 7.514 7.511 7.503 7.500 7.497 7.493 7.485 7.467 7.461 7.452 7.448 7.444 7.441 7.423 7.402 7.397 7.386 7.382 7.378 7.368 7.364 7.361 7.253 6.374 5.093 5.080 0.001
4h
1.00 1.00 1.02 1.98 6.92 0.95 2.00
f1 (ppm)

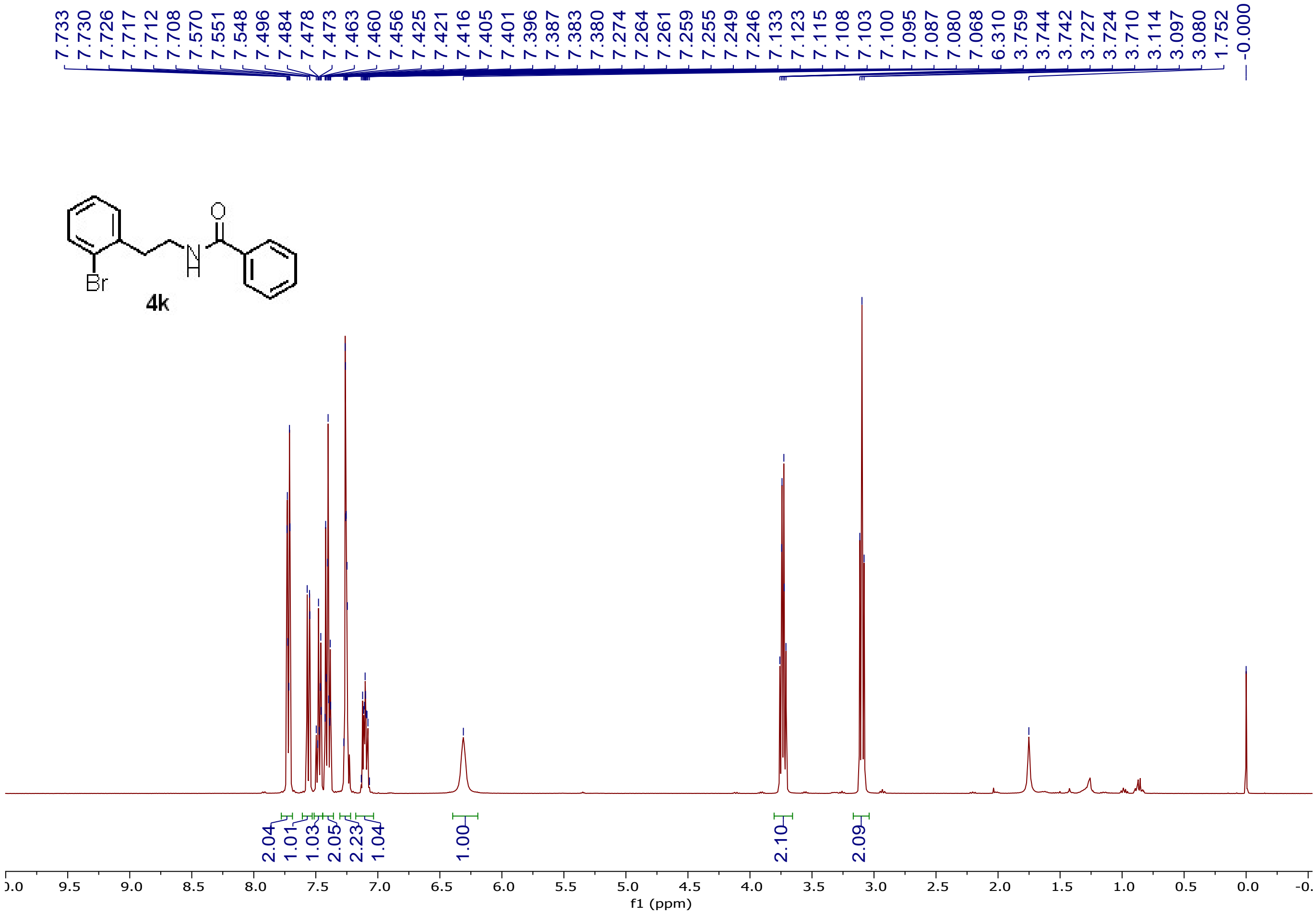

167.137
134.286 133.918 133.357 131.511 131.482 128.799 128.776 128.544 126.936 126.755 126.048 125.403 123.488
77.317 77.001 76.682
42.407
4h
f1 (ppm)



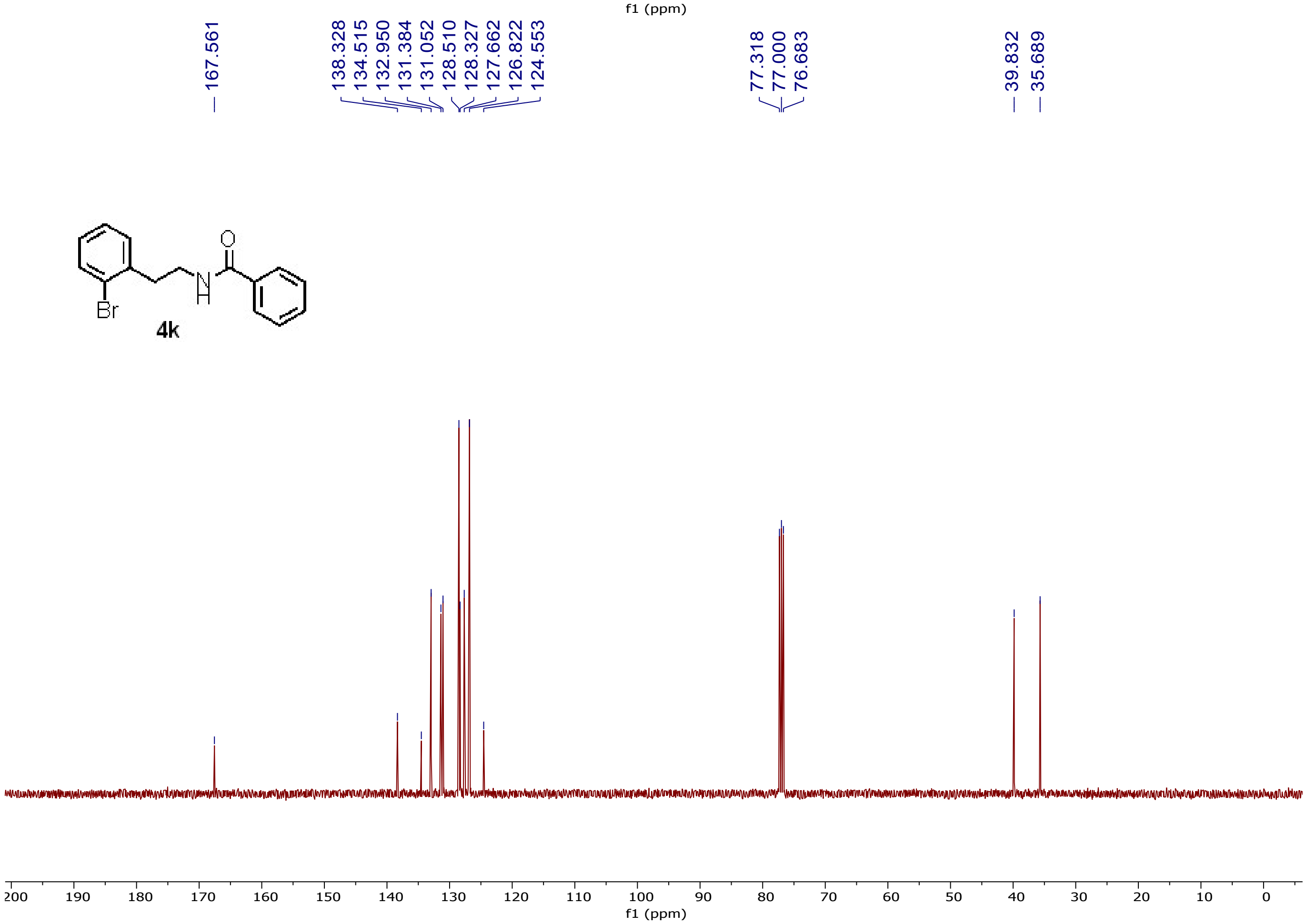



8.528
8.520
7.792
7.774
7.718
7.623
7.618
7.604
7.599
7.584
7.580
7.466
7.463
7.455
7.448
7.442
7.434
7.430
7.426
7.400
7.385
7.380
7.367
7.363
7.359
7.194
7.174
7.156
7.144
7.137
7.125
3.857
3.842
3.827
3.811
3.100
3.084
3.069
-0.000
N
N
H
O
4j
0.96
2.00
0.90
1.05
1.02
1.95
1.96
2.00
2.77
f1 (ppm)

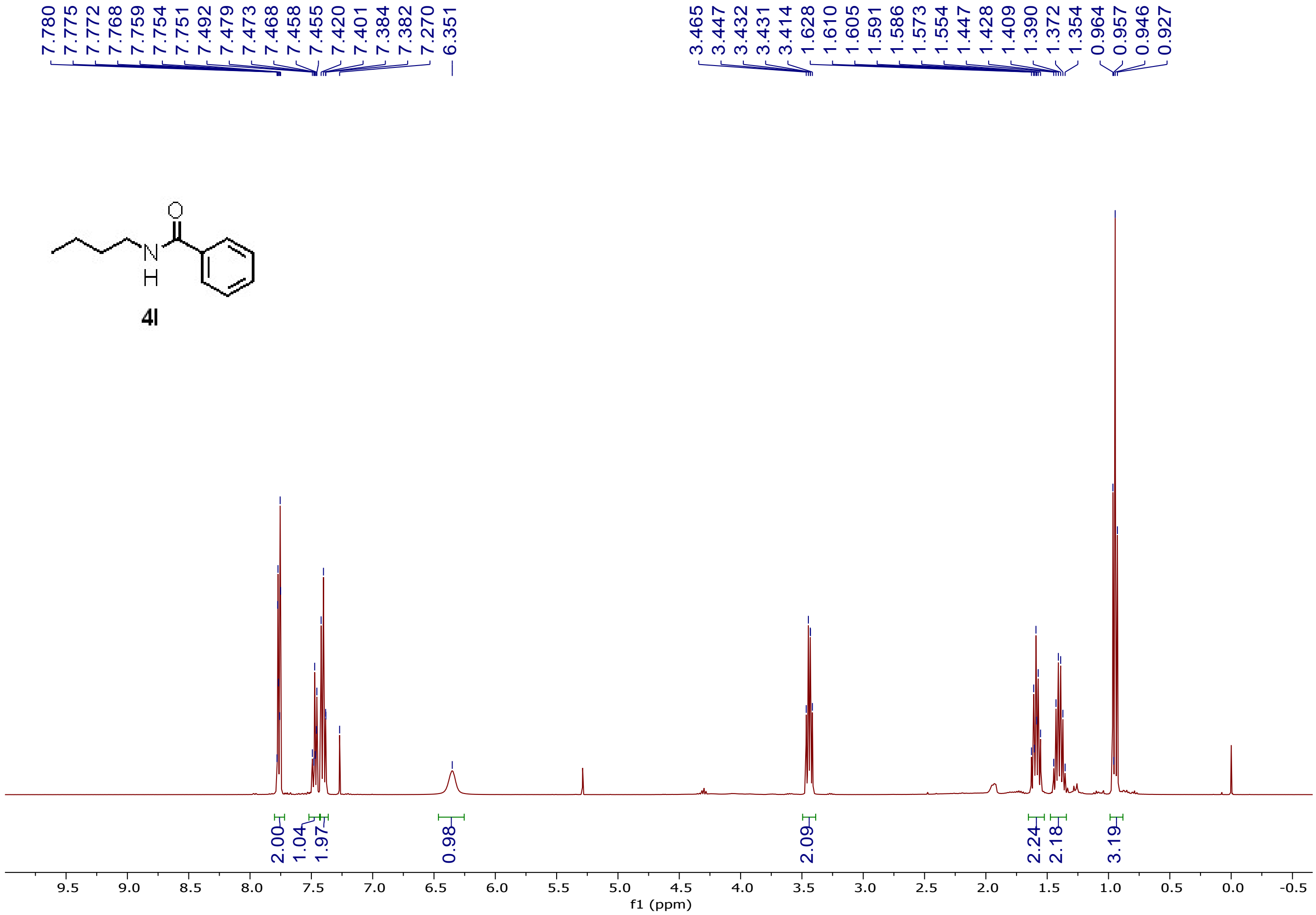

167.227
159.552
148.895
136.621
134.544
131.052
128.262
126.792
123.391
121.491
77.318
77.000
76.681
39.153
36.530
N
N
H
O
4j
f1 (ppm)

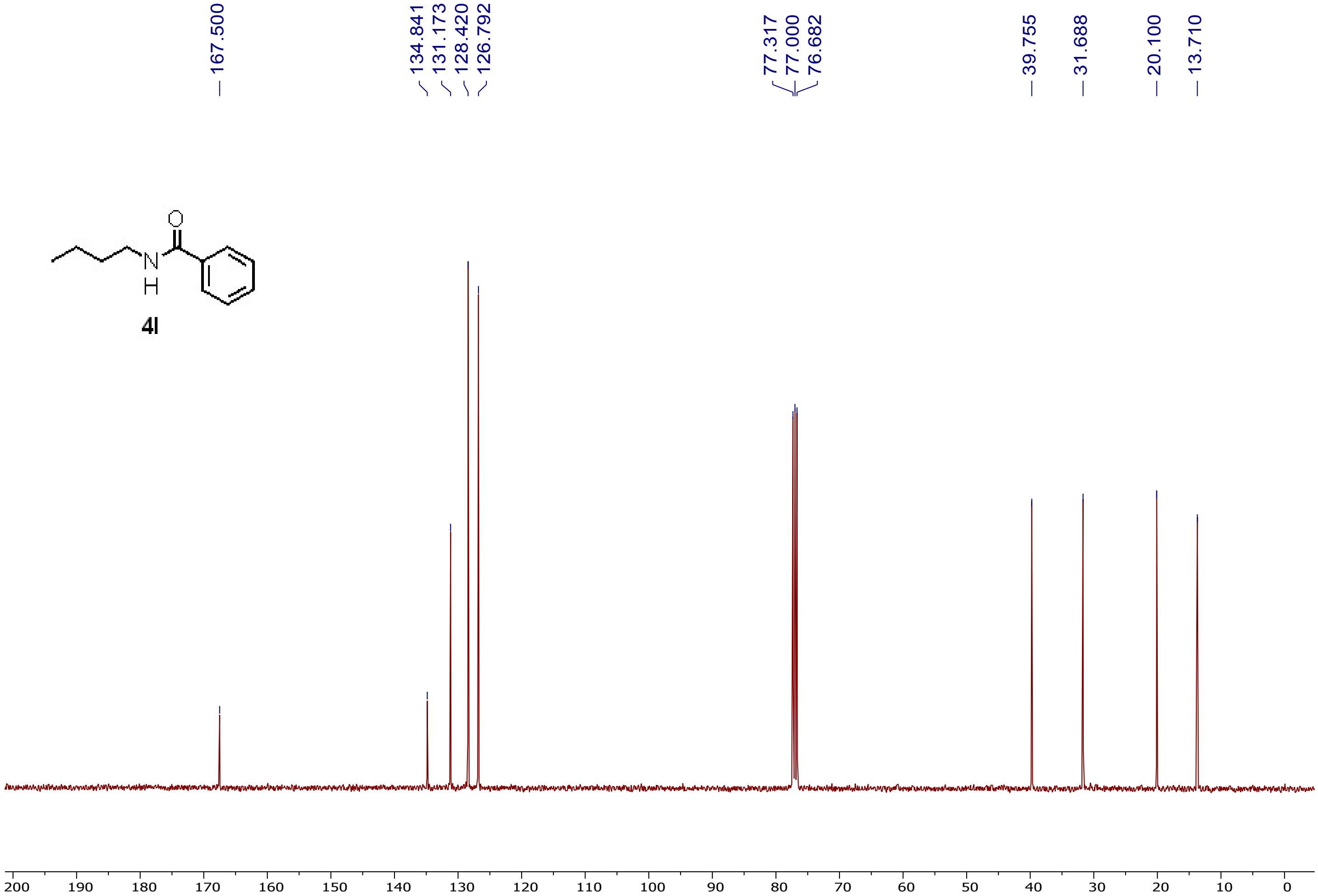


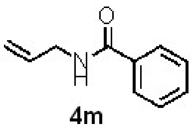
7.733 7.730 7.726 7.717 7.712 7.708 7.570 7.551 7.548 7.496 7.484 7.478 7.473 7.463 7.460 7.456 7.425 7.421 7.416 7.405 7.401 7.396 7.387 7.383 7.380 7.274 7.264 7.261 7.259 7.255 7.249 7.246 7.133 7.123 7.115 7.108 7.103 7.100 7.095 7.087 7.080 7.068 6.310 3.759 3.744 3.742 3.727 3.724 3.710 3.114 3.097 3.080 1.752 -0.000
Br
4k
2.04 1.01 1.03 2.05 2.23 1.04 1.00 2.10 2.09
f1 (ppm)



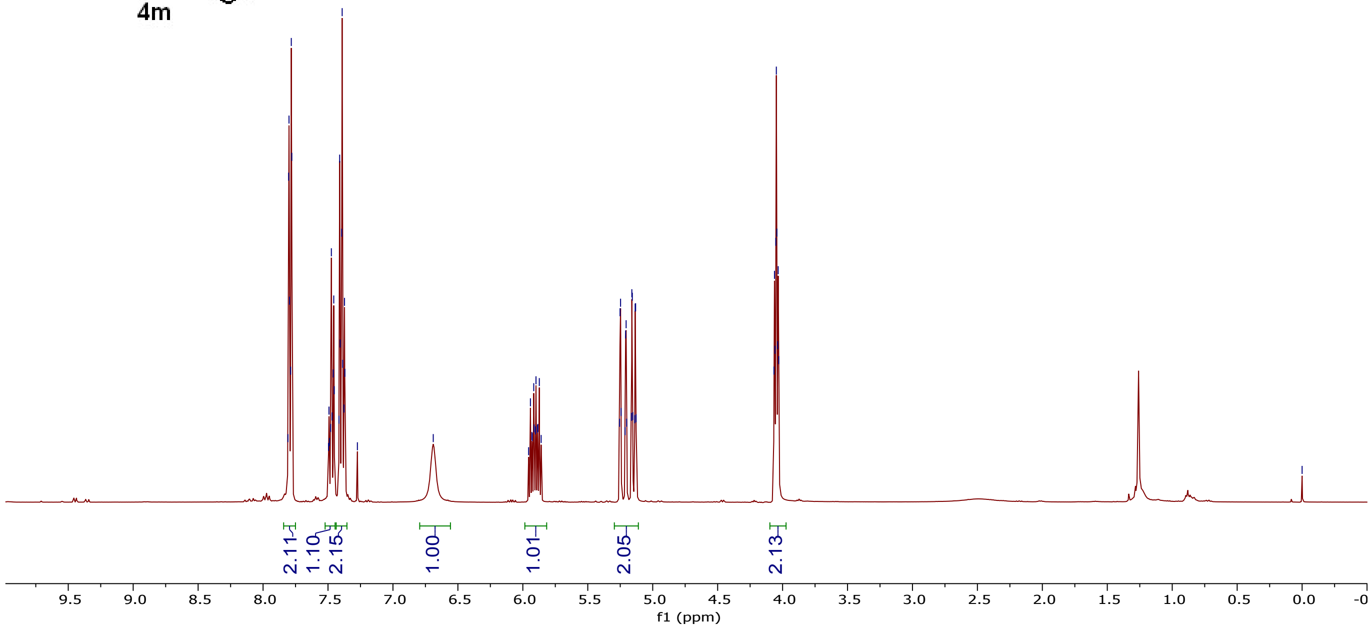
167.561
138.328 134.515 132.950 131.384 131.052 128.510 128.327 127.662 126.822 124.553
77.318 77.000 76.683
39.832 35.689
Br
4k
f1 (ppm)

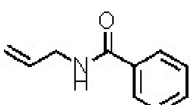

7.780 7.775 7.772 7.768 7.759 7.754 7.751 7.492 7.479 7.473 7.468 7.458 7.455 7.420 7.401 7.384 7.382 7.270 6.351
3.465 3.447 3.432 3.431 3.414 1.628 1.610 1.605 1.591 1.586 1.573 1.554 1.447 1.428 1.409 1.390 1.372 1.354 0.964 0.957 0.946 0.927
O
N
H
4l
2.00 1.04 1.97 0.98 2.09 2.24 2.18 3.19
f1 (ppm)


167.500
134.841 131.173 128.420 126.792
77.317 77.000 76.682
39.755 31.688 20.100 13.710
O
N
H
4l
f1 (ppm)

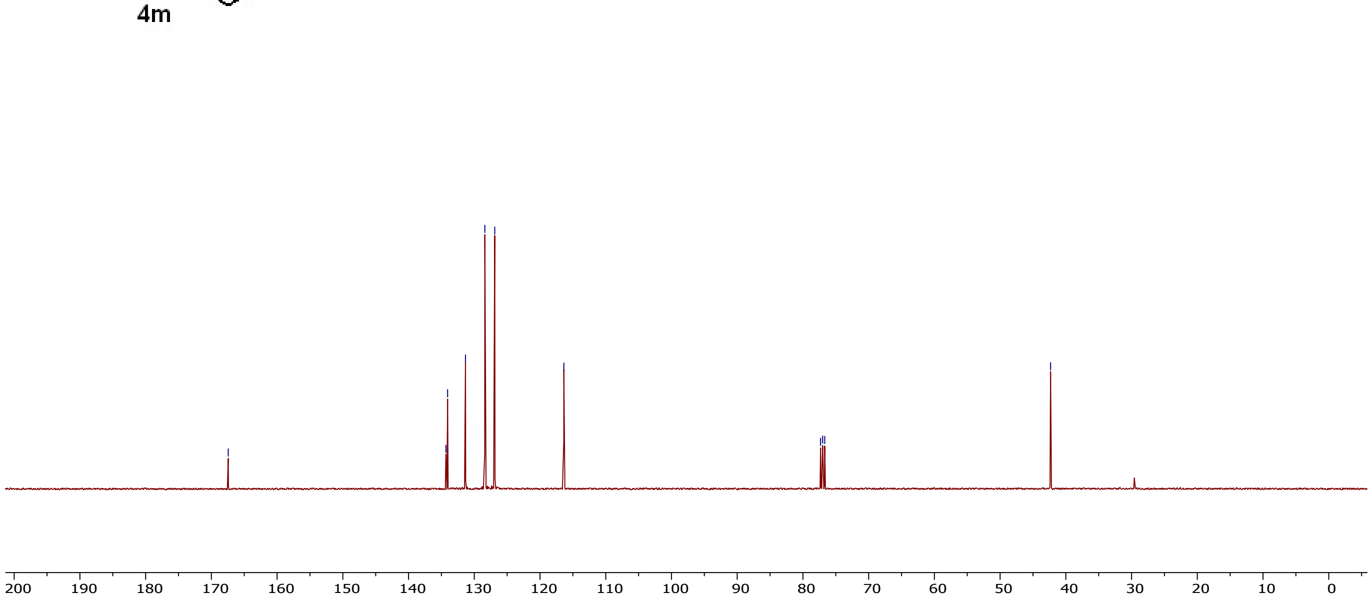


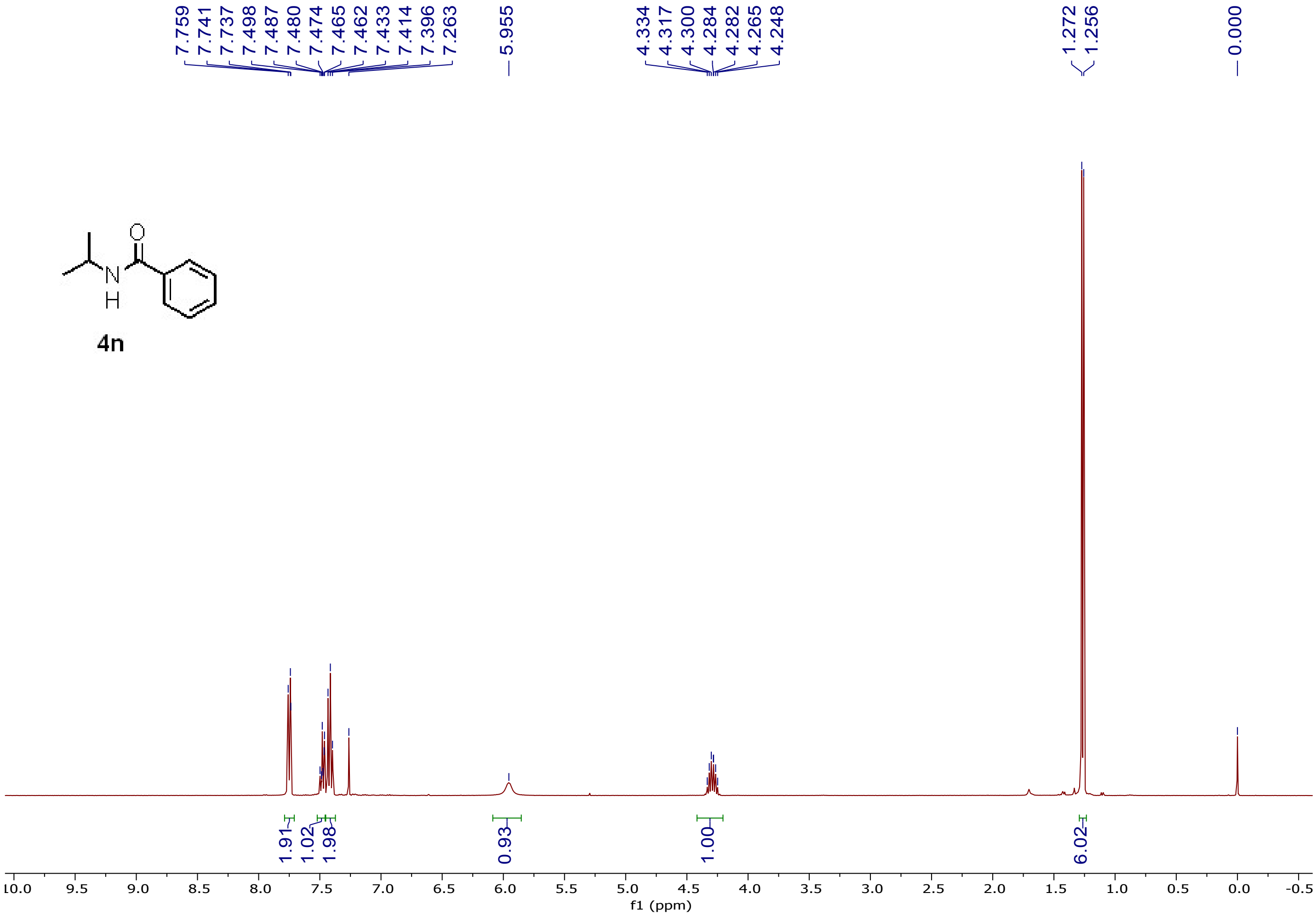
4m

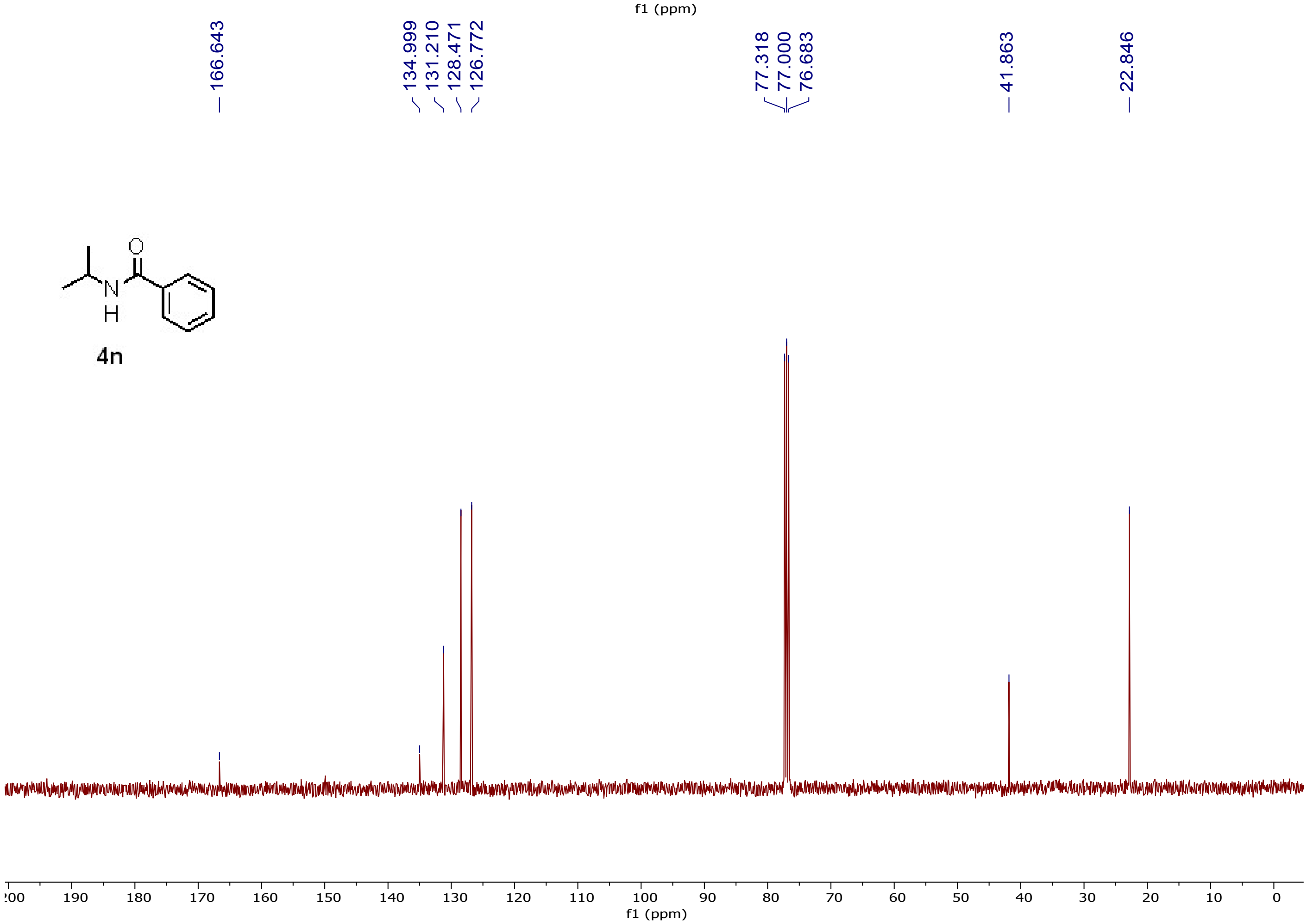

7.803
7.800
7.796
7.788
7.783
7.779
7.492
7.480
7.474
7.469
7.459
7.456
7.452
7.415
7.410
7.406
7.395
7.391
7.387
7.377
7.373
7.370
6.690
5.942
5.930
5.927
5.916
5.913
5.902
5.899
5.887
5.885
5.873
5.255
5.251
5.248
5.243
5.213
5.209
5.205
5.201
5.164
5.161
5.157
5.153
5.139
5.135
5.132
5.128
4.066
4.062
4.059
4.052
4.048
4.044
4.038
4.034
4.030
2.11
1.10
2.15
1.00
1.01
2.05
2.13
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

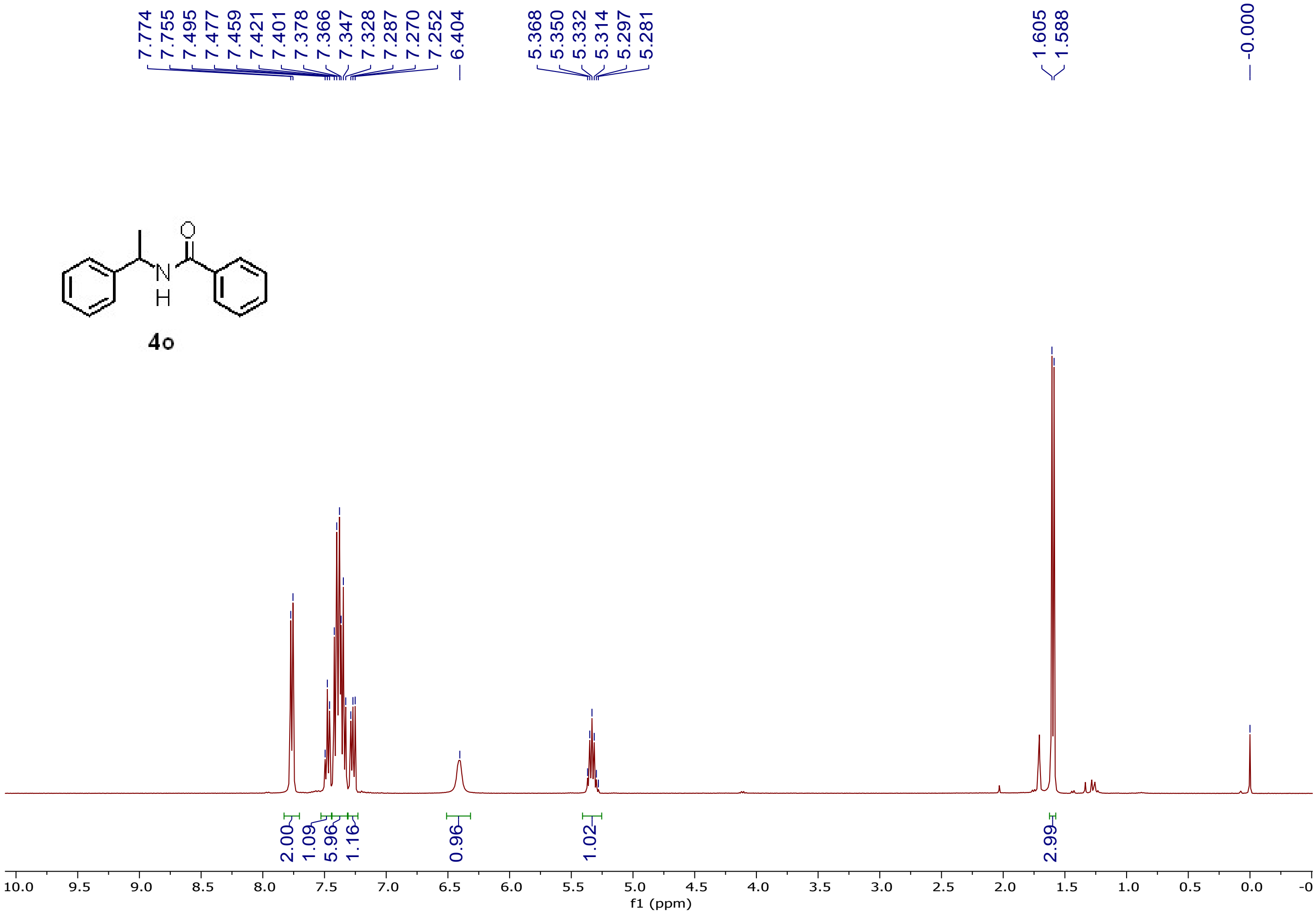


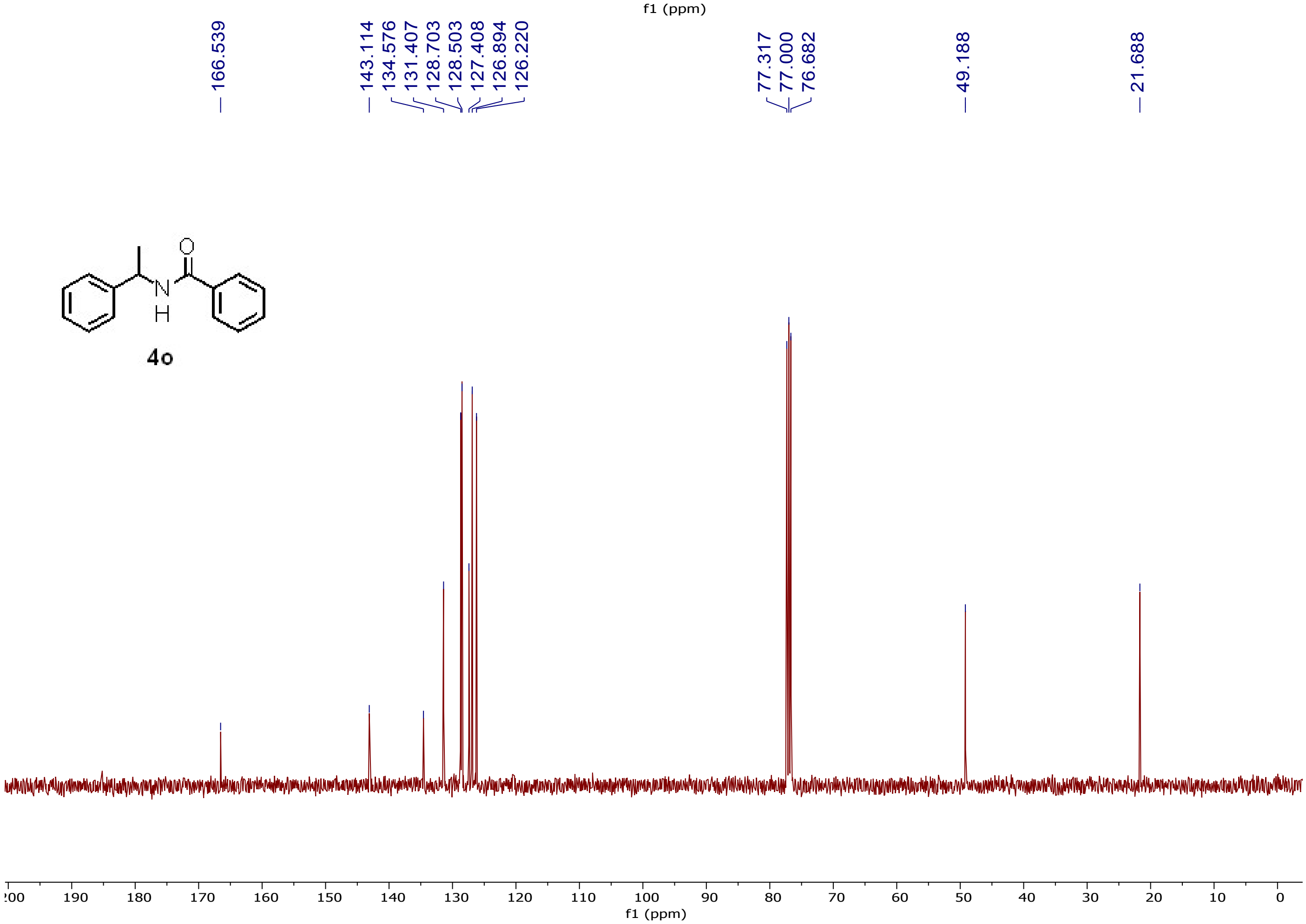
4m

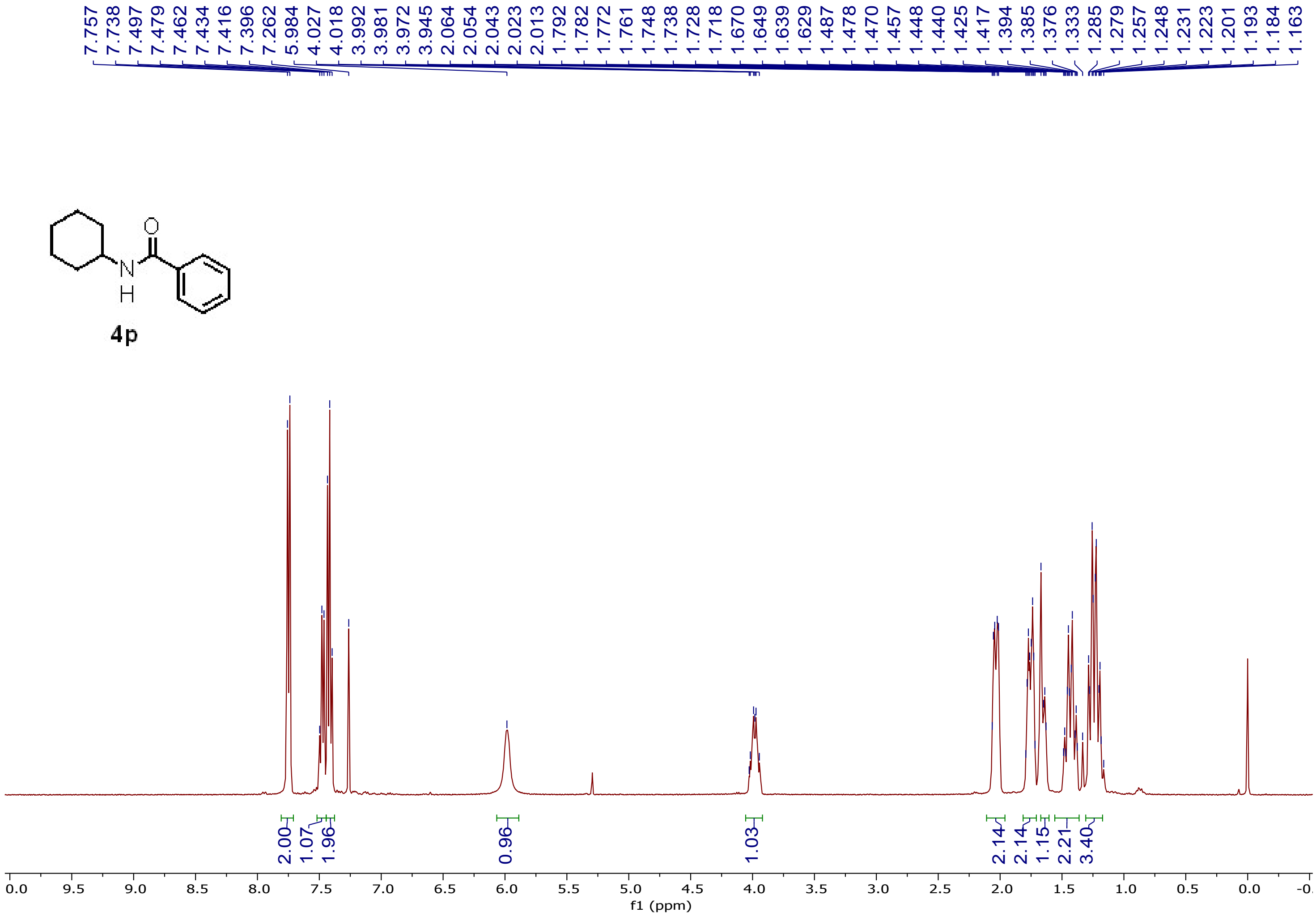

167.431
134.317
134.059
131.338
128.395
126.886
116.374
77.318
77.000
76.682
42.322
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

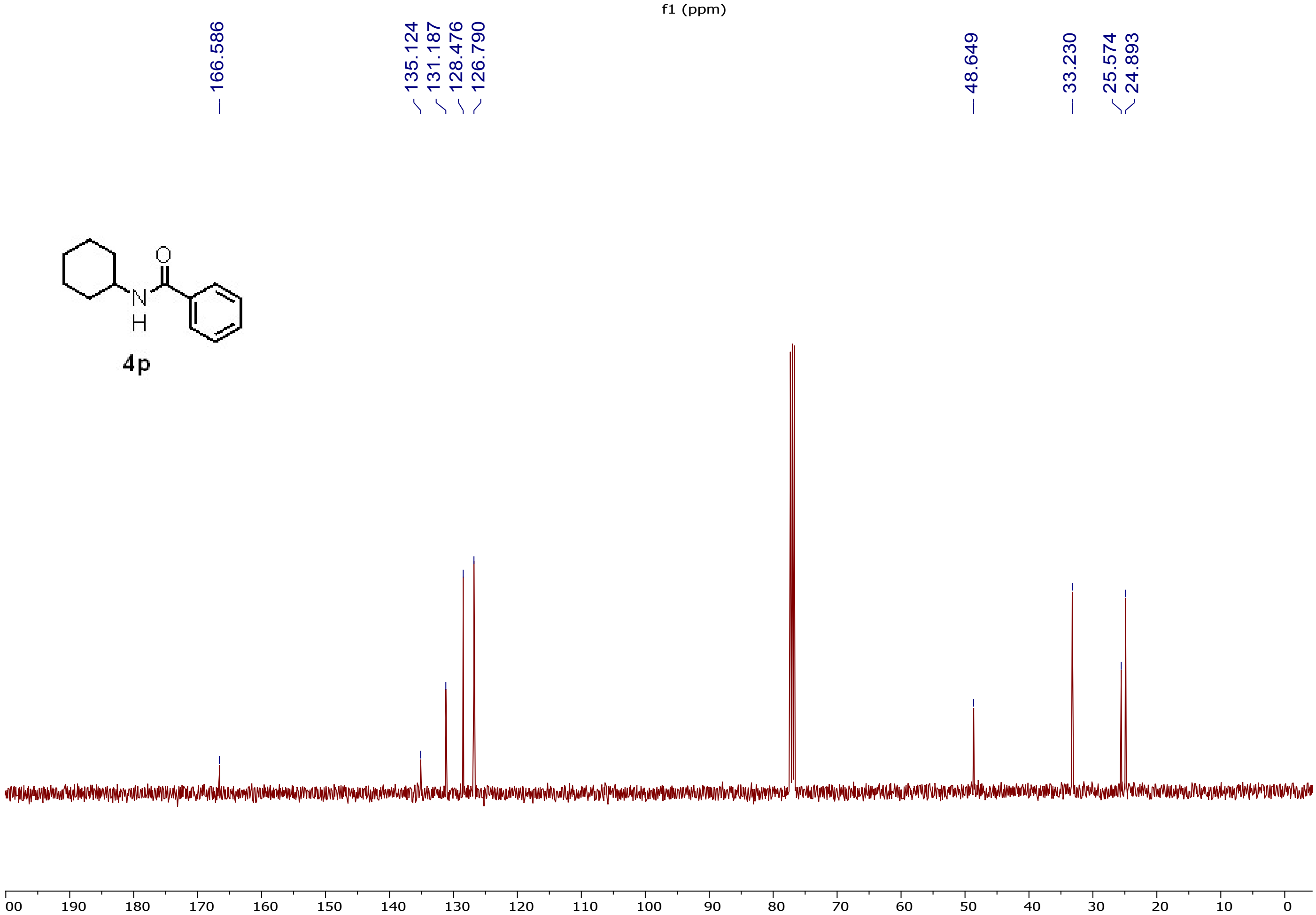

7.759 7.741 7.737 7.498 7.487 7.480 7.474 7.465 7.462 7.433 7.414 7.396 7.263
5.955
4.334 4.317 4.300 4.284 4.282 4.265 4.248
1.272 1.256
0.000
4n
1.91 1.02 1.98 0.93 1.00 6.02
f1 (ppm)

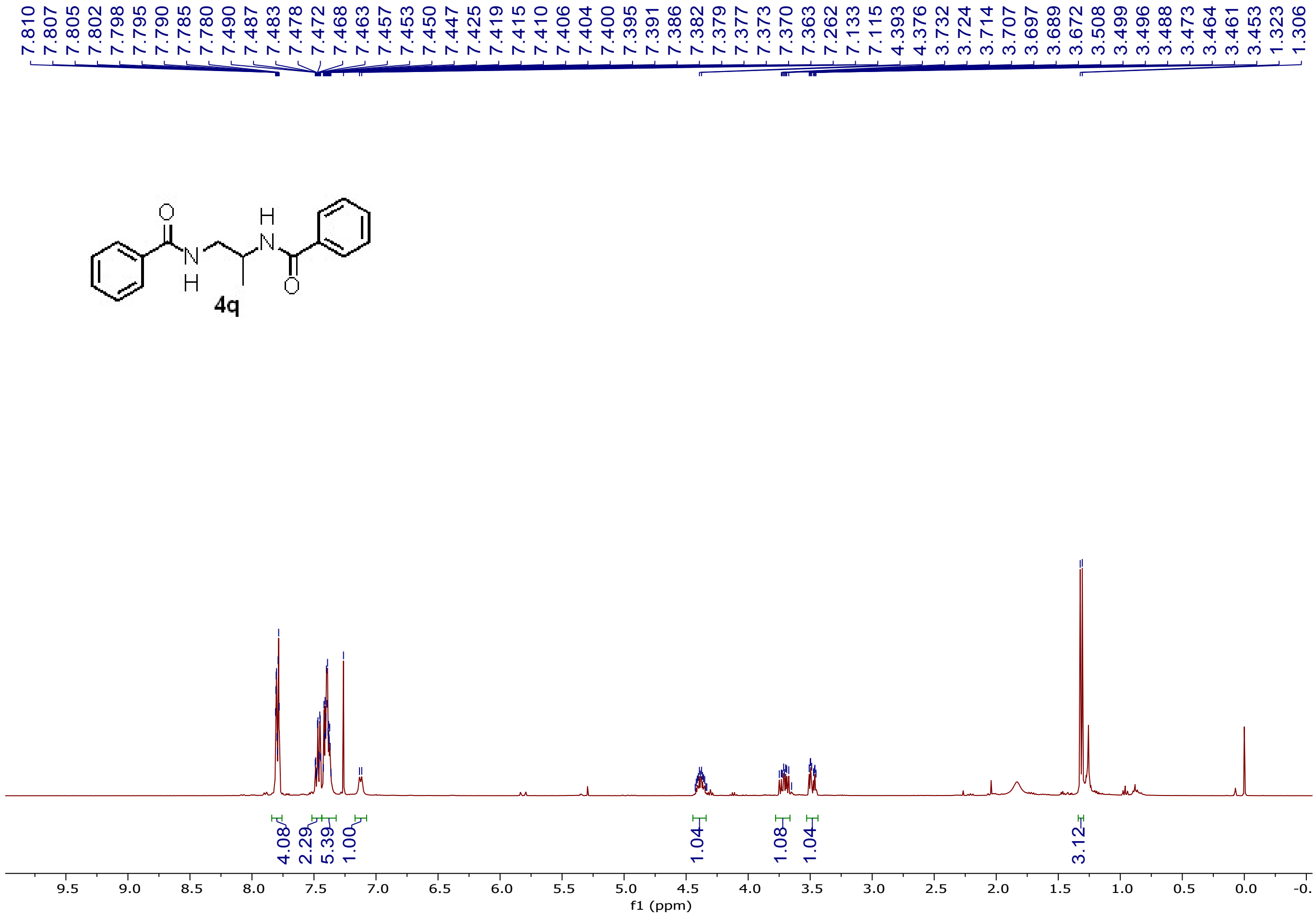

166.643
134.999 131.210 128.471 126.772
77.318 77.000 76.683
41.863
22.846
4n
f1 (ppm)

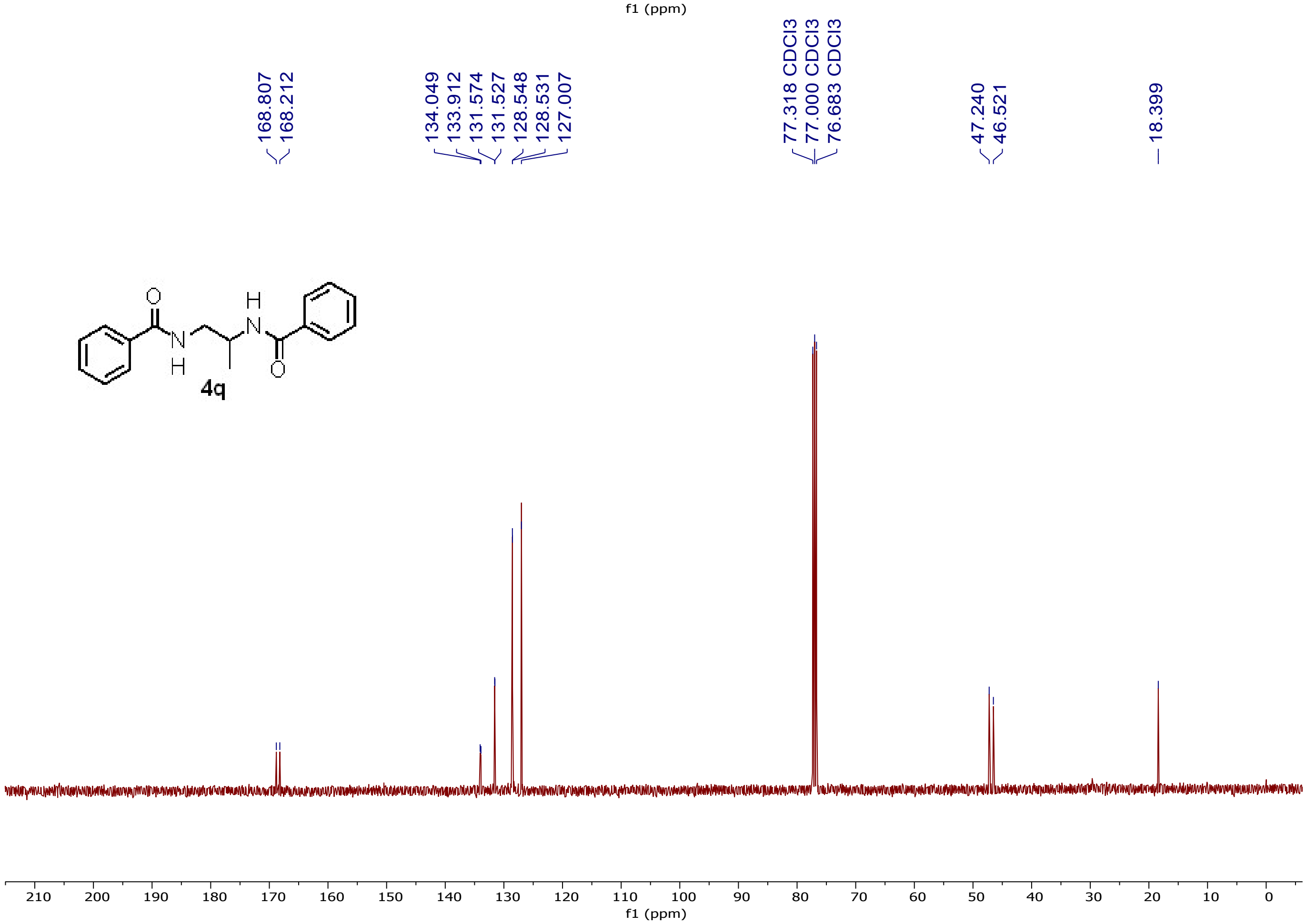

7.774
7.755
7.495
7.477
7.459
7.421
7.401
7.378
7.366
7.347
7.328
7.287
7.270
7.252
6.404
5.368
5.350
5.332
5.314
5.297
5.281
1.605
1.588
-0.000
4o
2.00
1.09
5.96
1.16
0.96
1.02
2.99
f1 (ppm)

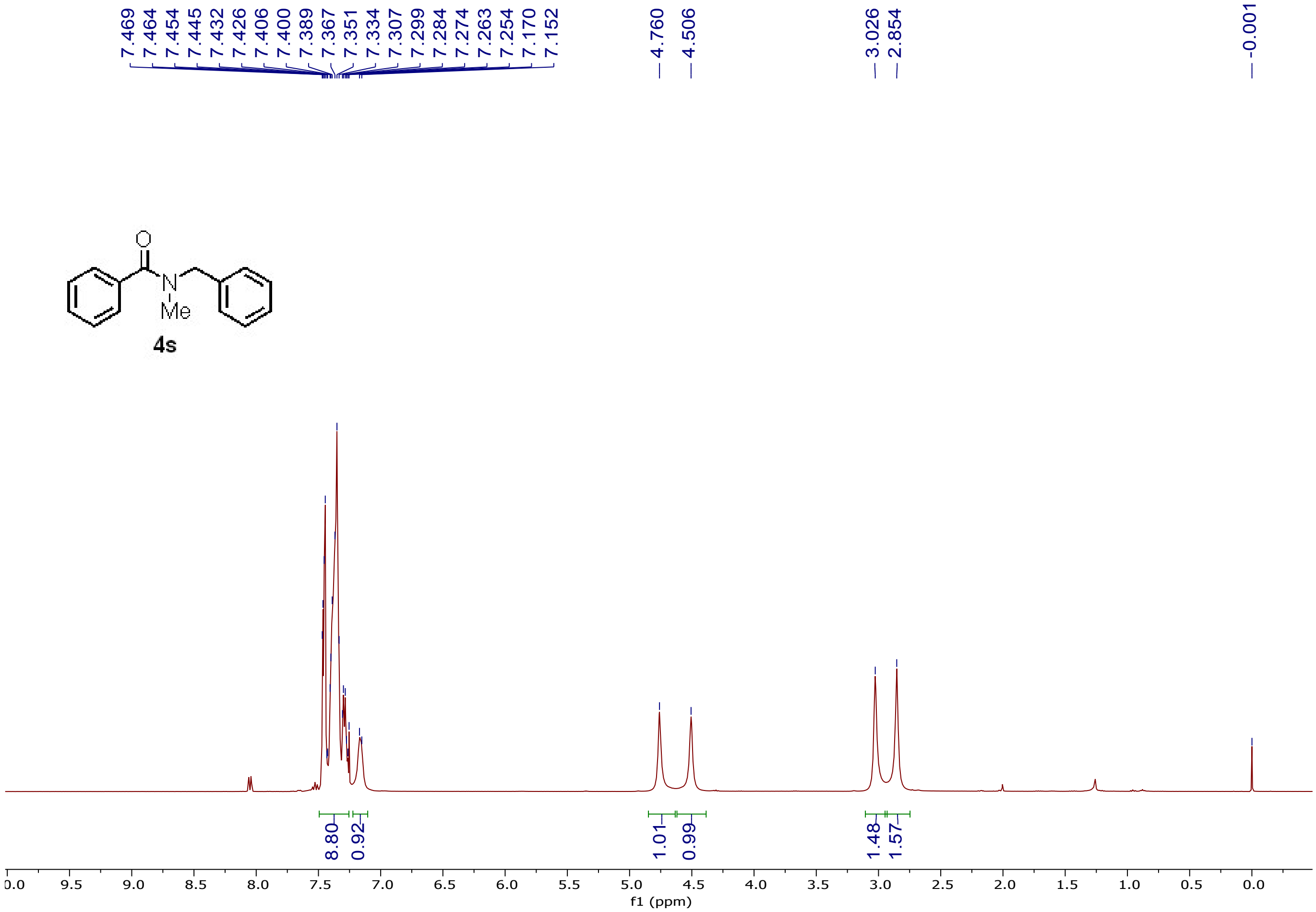

166.539
143.114
134.576
131.407
128.703
128.503
127.408
126.894
126.220
77.317
77.000
76.682
49.188
21.688
4o
f1 (ppm)

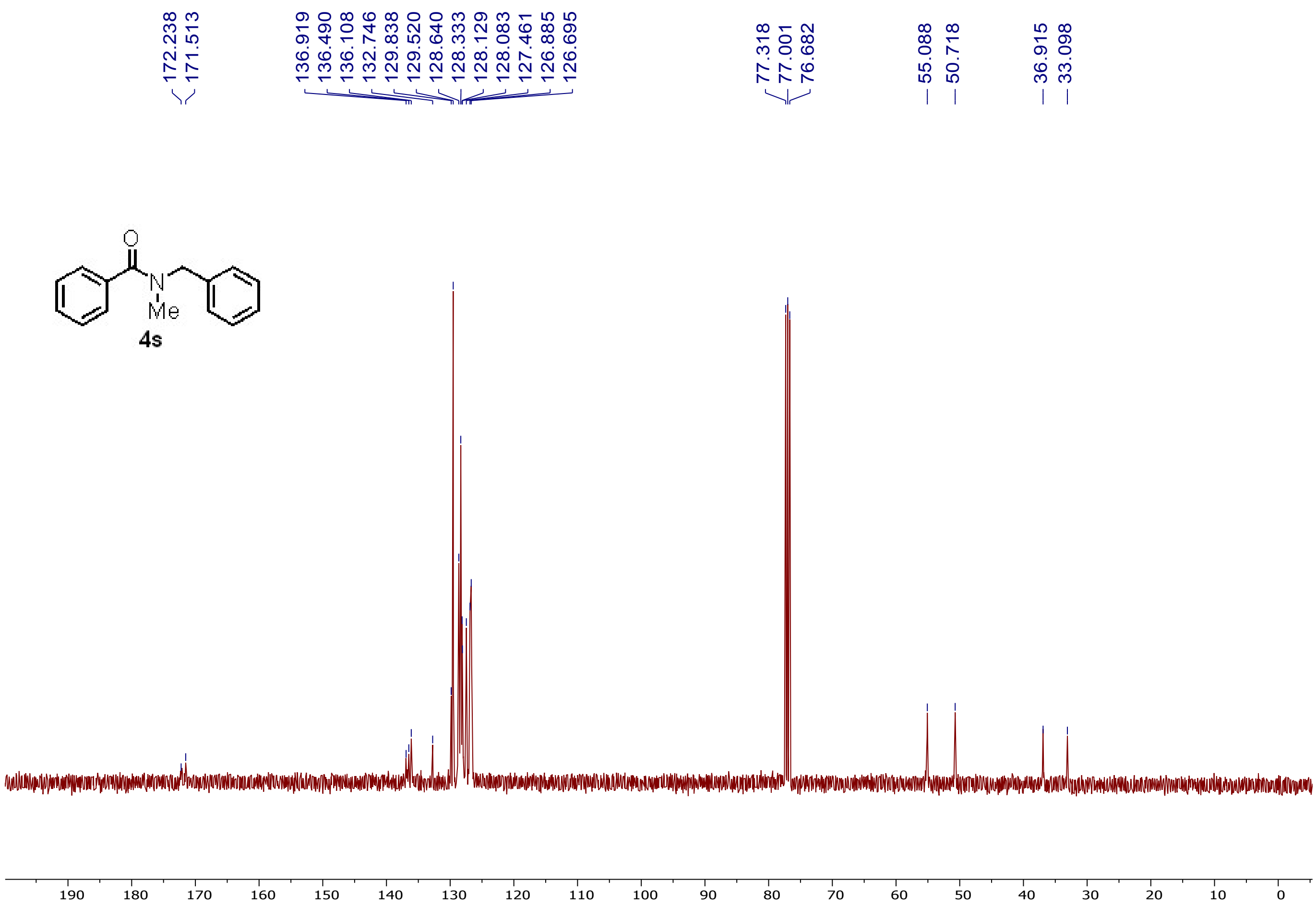

7.757 7.738 7.497 7.479 7.462 7.434 7.416 7.396 7.262 5.984 4.027 4.018 3.992 3.981 3.972 3.945 2.064 2.054 2.043 2.023 2.013 1.792 1.782 1.772 1.761 1.748 1.738 1.728 1.718 1.670 1.649 1.639 1.629 1.487 1.478 1.470 1.457 1.448 1.440 1.425 1.417 1.394 1.385 1.376 1.333 1.285 1.279 1.257 1.248 1.231 1.223 1.201 1.193 1.184 1.163
4p
2.00 1.07 1.96 0.96 1.03 2.14 2.14 1.15 2.21 3.40
f1 (ppm)

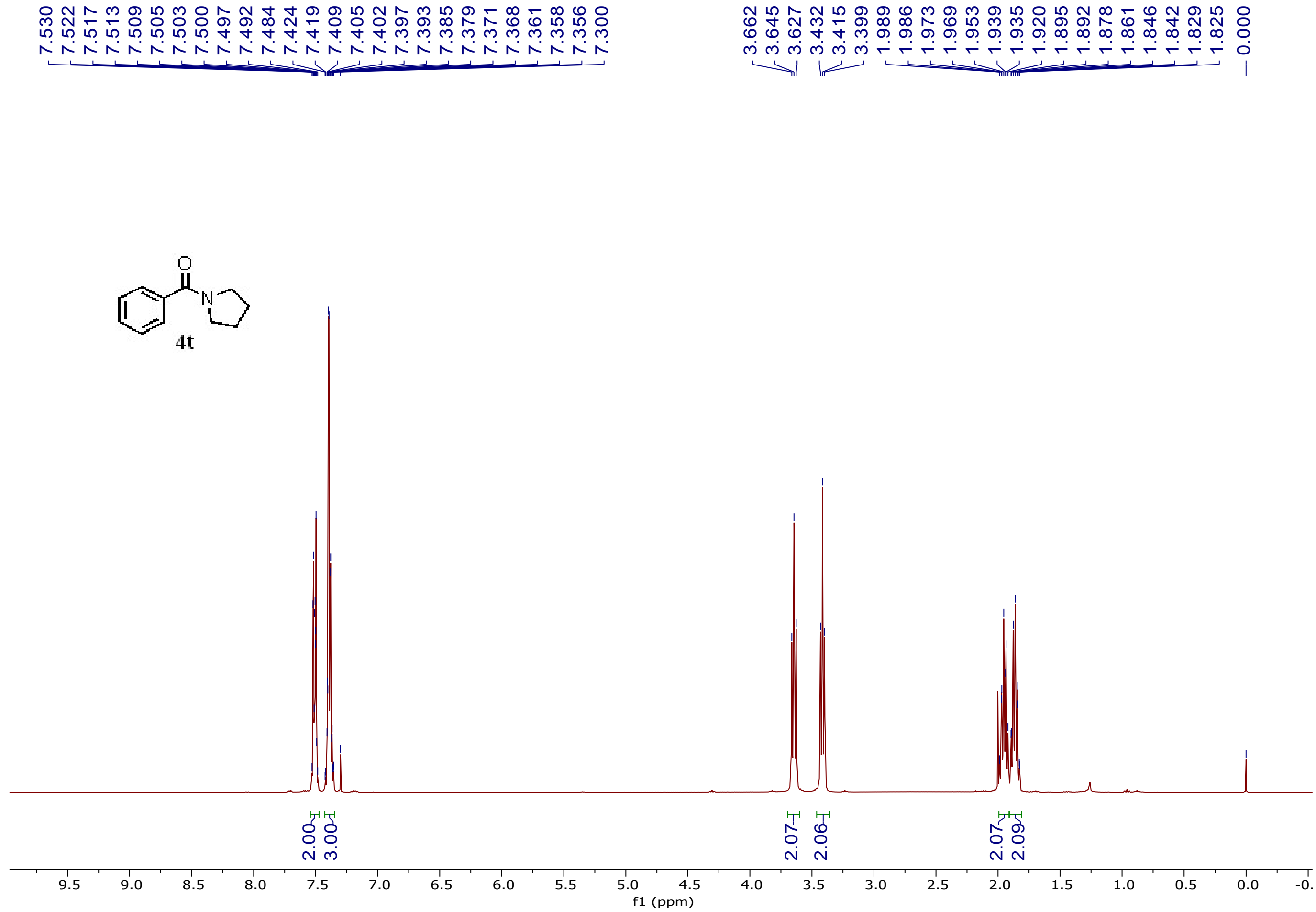

166.586
135.124 131.187 128.476 126.790
48.649
33.230
25.574 24.893
4p
f1 (ppm)

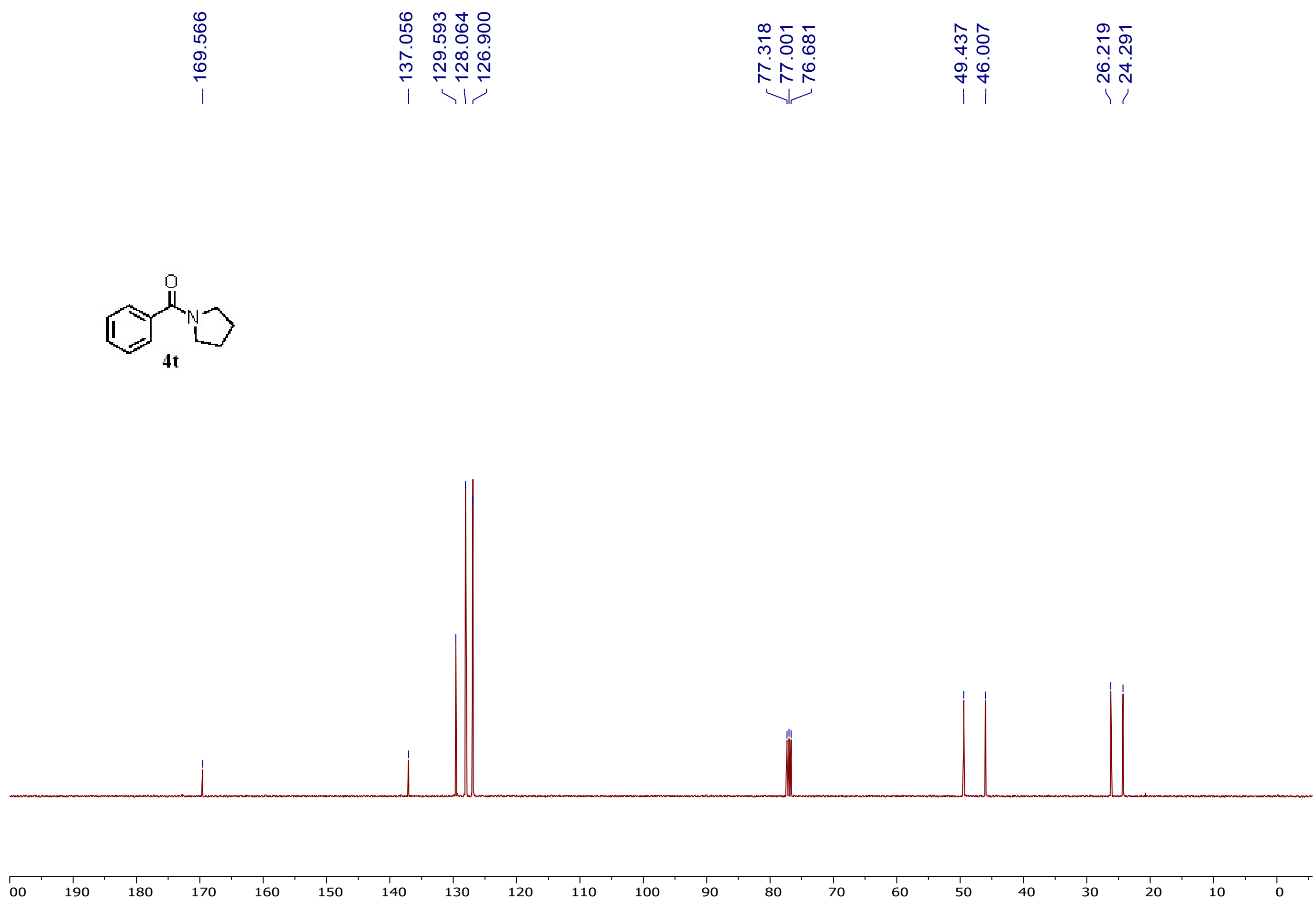

7.810 7.807 7.805 7.802 7.798 7.795 7.790 7.785 7.780 7.490 7.487 7.483 7.478 7.472 7.468 7.463 7.457 7.453 7.450 7.447 7.425 7.419 7.415 7.410 7.406 7.404 7.400 7.395 7.391 7.386 7.382 7.379 7.377 7.373 7.370 7.363 7.262 7.133 7.115 4.393 4.376 3.732 3.724 3.714 3.707 3.697 3.689 3.672 3.508 3.499 3.496 3.488 3.473 3.464 3.461 3.453 1.323 1.306
4q
4.08 2.29 5.39 1.00 1.04 1.08 1.04 3.12
f1 (ppm)

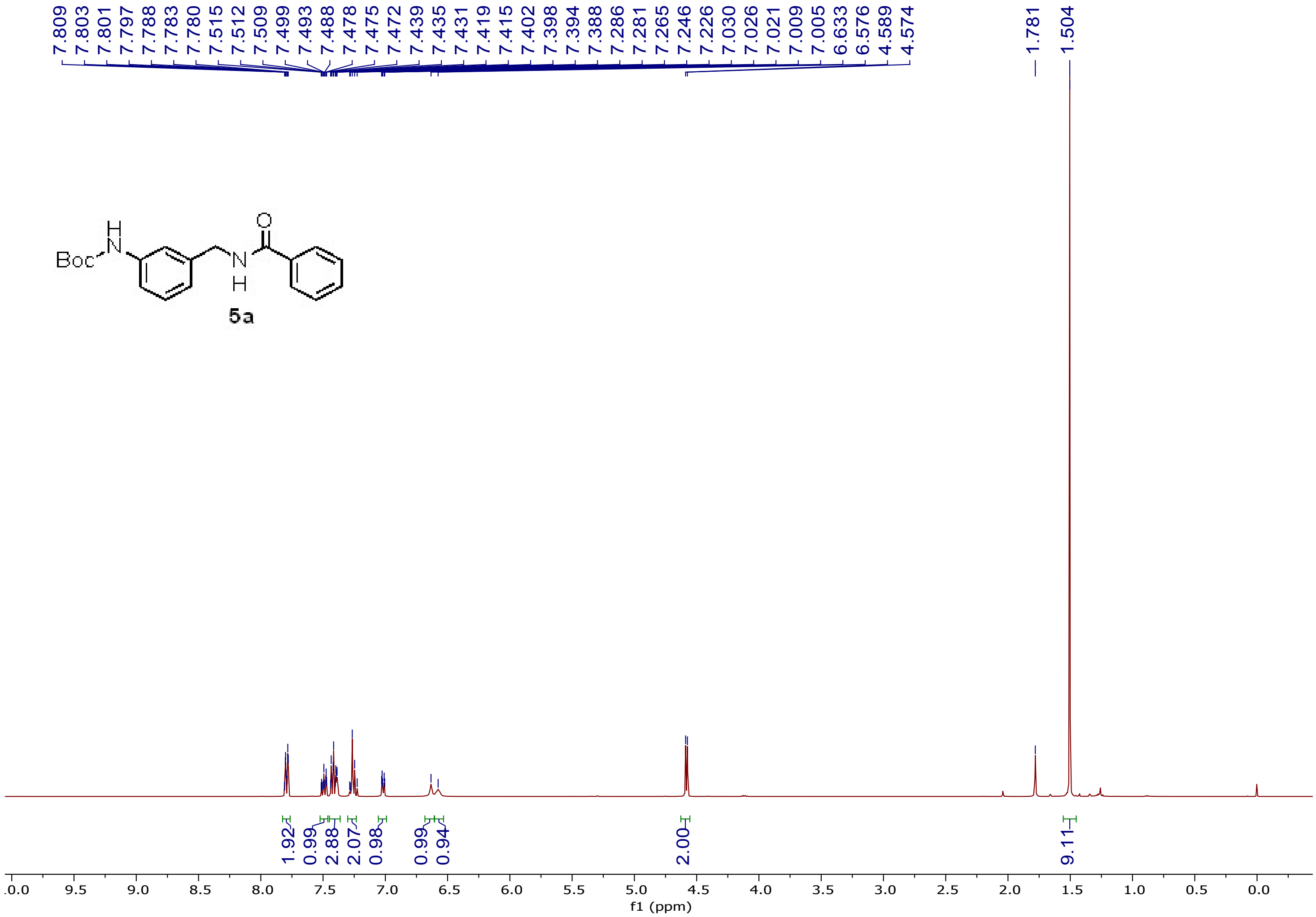

168.807 168.212 134.049 133.912 131.574 131.527 128.548 128.531 127.007 77.318 CDCl3 77.000 CDCl3 76.683 CDCl3 47.240 46.521 18.399
4q
f1 (ppm)

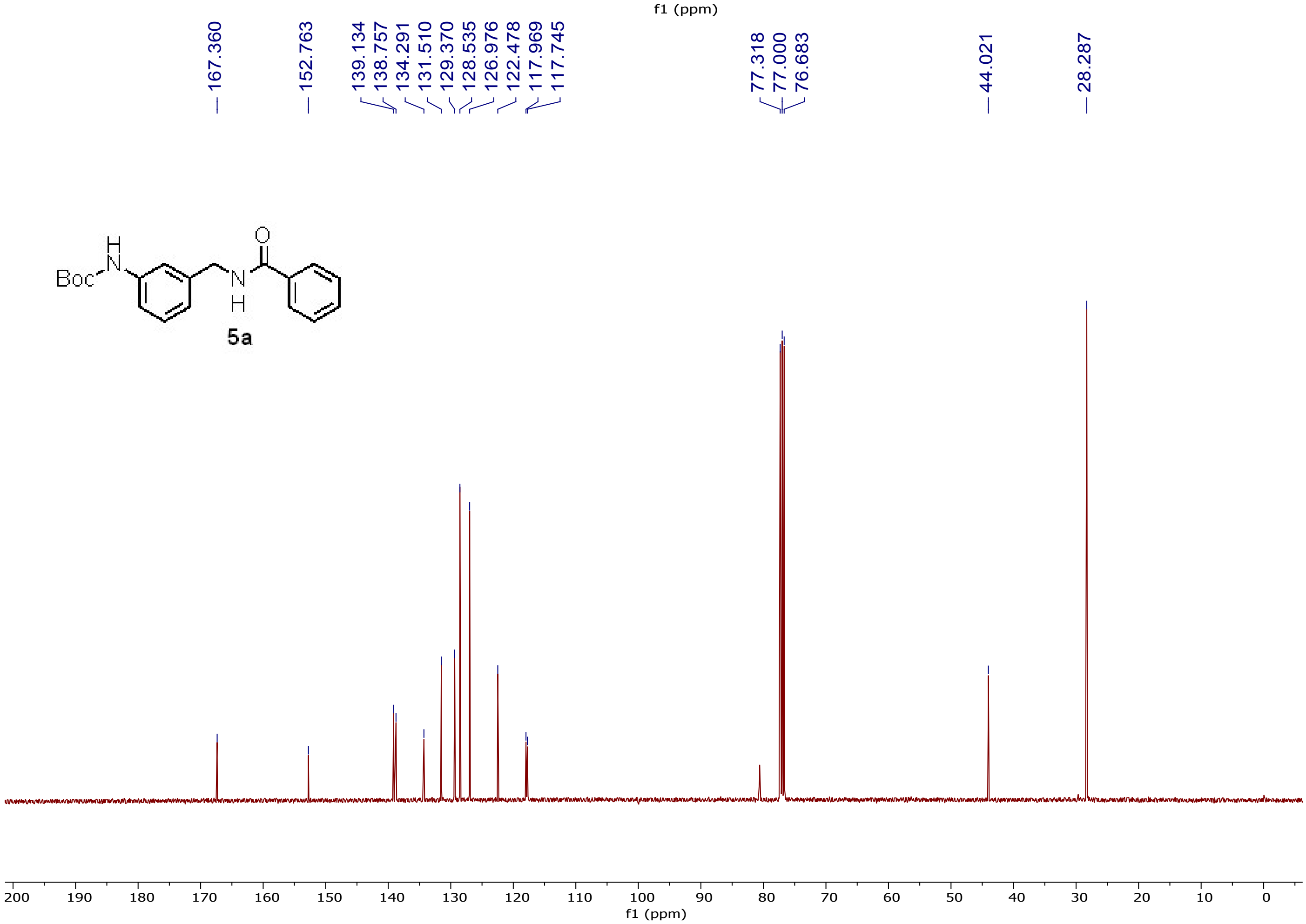

7.469
7.464
7.454
7.445
7.432
7.426
7.406
7.400
7.389
7.367
7.351
7.334
7.307
7.299
7.284
7.274
7.263
7.254
7.170
7.152
4.760
4.506
3.026
2.854
-0.001
O
N
Me
4s
8.80
0.92
1.01
0.99
1.48
1.57
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

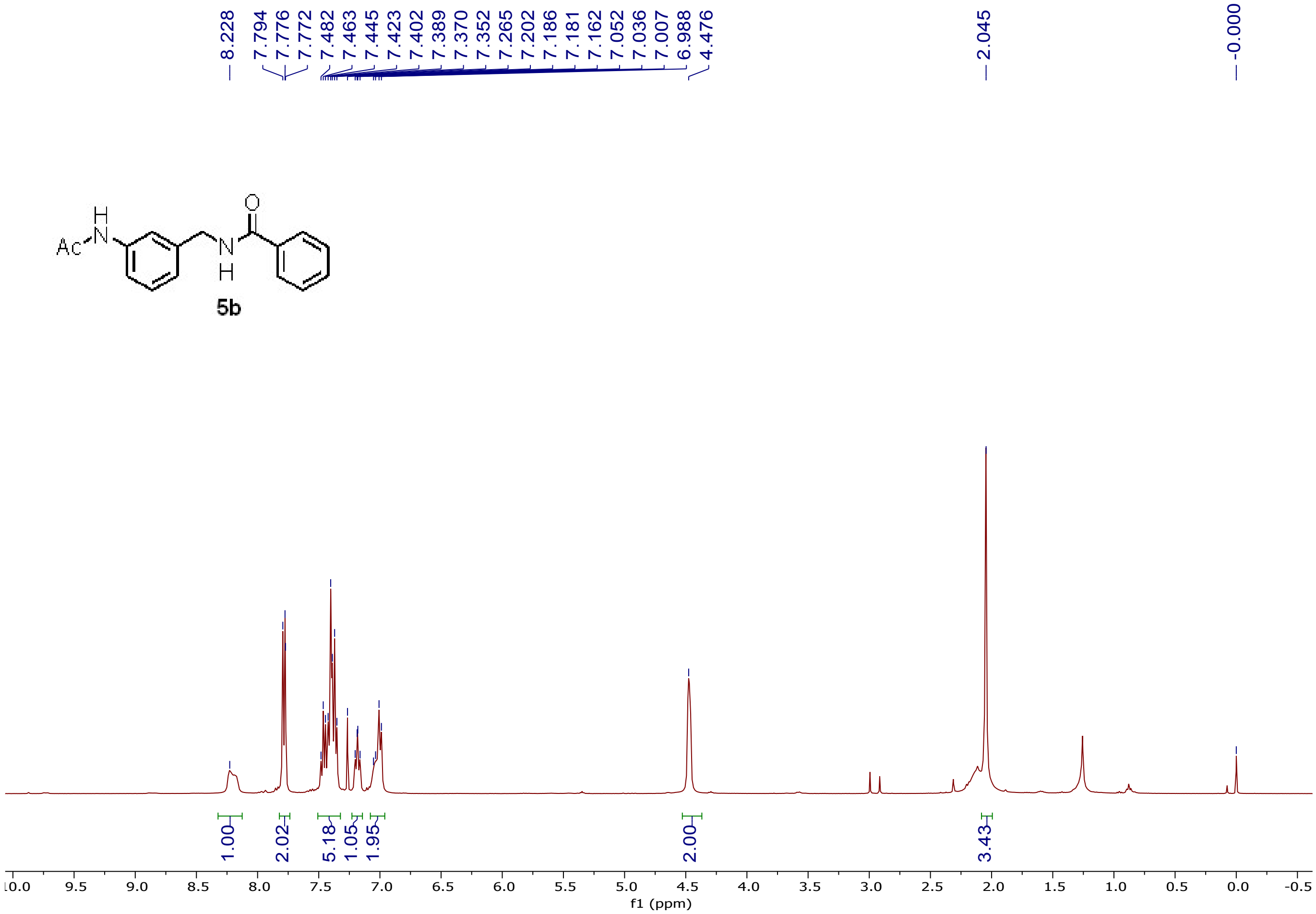

172.238
171.513
136.919
136.490
136.108
132.746
129.838
129.520
128.640
128.333
128.129
128.083
127.461
126.885
126.695
77.318
77.001
76.682
55.088
50.718
36.915
33.098
O
N
Me
4s
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

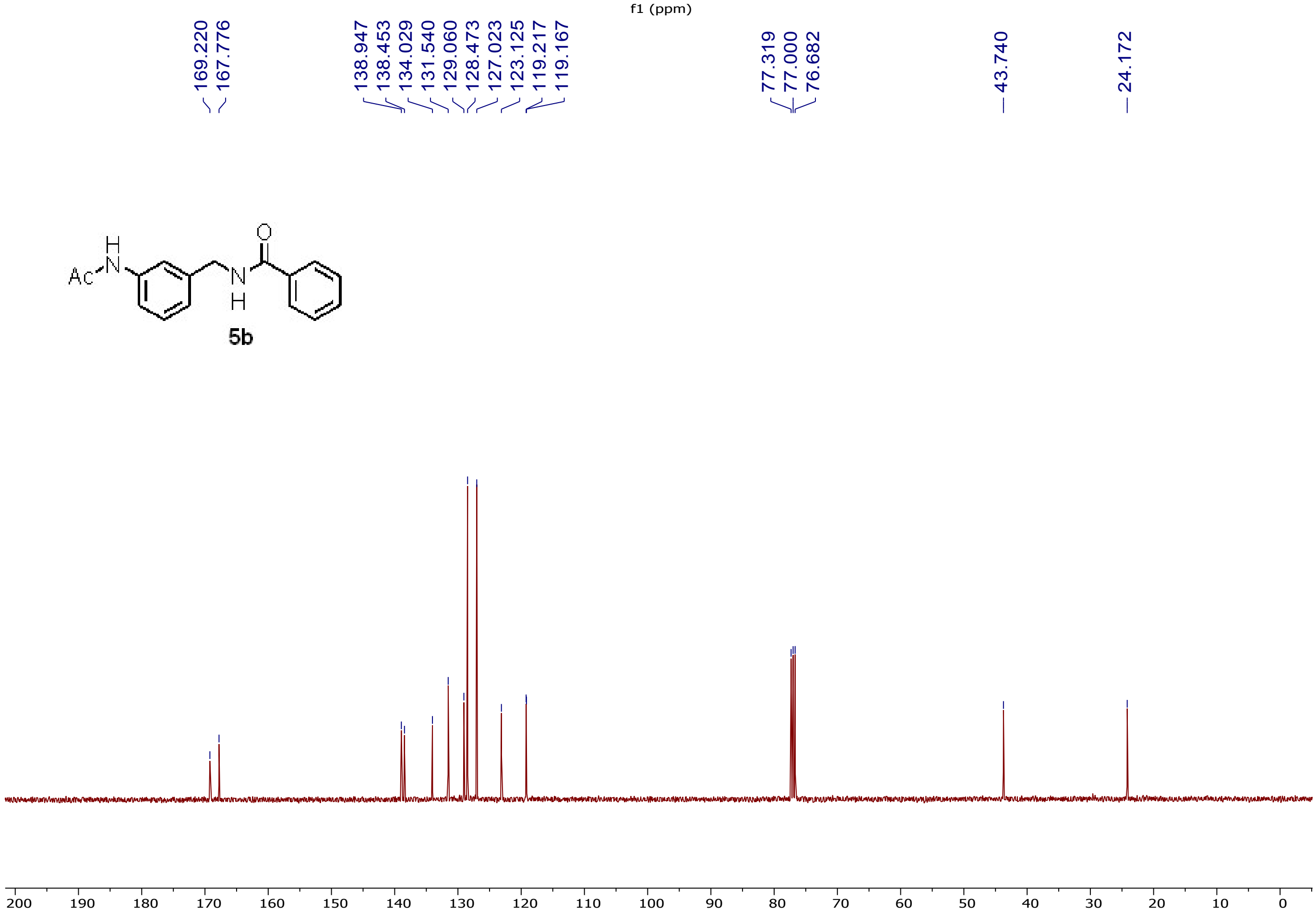

7.530
7.522
7.517
7.513
7.509
7.505
7.503
7.500
7.497
7.492
7.484
7.424
7.419
7.409
7.405
7.402
7.397
7.393
7.385
7.379
7.371
7.368
7.361
7.358
7.356
7.300
3.662
3.645
3.627
3.432
3.415
3.399
1.989
1.986
1.973
1.969
1.953
1.939
1.935
1.920
1.895
1.892
1.878
1.861
1.846
1.842
1.829
1.825
0.000
4t
2.00
3.00
2.07
2.06
2.07
2.09
f1 (ppm)


169.566
137.056
129.593
128.064
126.900
77.318
77.001
76.681
49.437
46.007
26.219
24.291
4t
f1 (ppm)


7.809 7.803 7.801 7.797 7.788 7.783 7.780 7.515 7.512 7.509 7.499 7.493 7.488 7.478 7.475 7.472 7.439 7.435 7.431 7.419 7.415 7.402 7.398 7.394 7.388 7.286 7.281 7.265 7.246 7.226 7.030 7.026 7.021 7.009 7.005 6.633 6.576 4.589 4.574
1.781
1.504
Boc
N
H
O
5a
1.92 0.99 2.88 2.07 0.98 0.99 0.94
2.00
9.11
f1 (ppm)


167.360
152.763
139.134 138.757 134.291 131.510 129.370 128.535 126.976 122.478 117.969 117.745
77.318 77.000 76.683
44.021
28.287
Boc
N
H
O
5a
f1 (ppm)


8.228
7.794
7.776
7.772
7.482
7.463
7.445
7.423
7.402
7.389
7.370
7.352
7.265
7.202
7.186
7.181
7.162
7.052
7.036
7.007
6.988
4.476
2.045
-0.000
Ac
N
H
N
H
O
5b
1.00
2.02
5.18
1.05
1.95
2.00
3.43
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


169.220
167.776
138.947
138.453
134.029
131.540
129.060
128.473
127.023
123.125
119.217
119.167
77.319
77.000
76.682
43.740
24.172
Ac
N
H
N
H
O
5b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)